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LINER MATERIALS EXPOSED TO HAZARDOUS AND TOXIC WASTES

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16. ABSTRACT

This exploratory experimental research project was conducted (1975-1983) to assess the relative effectiveness and durability of a wide variety of liner materials when exposed to hazardous wastes under conditions that simulate different aspects of service in on-land waste storage and disposal facilities. The materials studied included compacted soil, polymer-treated bentonite-sand mixtures, soil cement, hydraulic asphalt concrete, sprayed-on asphalt, and 31 flexible polymeric membranes based on polyvinyl chloride, chlorinated polyethylene, chlorosulfonated polyethylene, ethylene propylene rubber, neoprene, butyl rubber, elasticized polyolefin, and polyester elastomer. Four semicrystalline polymeric sheetings (polybutylene, low-density polyethylene, high-density polyethylene, and polypropylene), though not compounded for use as liners, were included in the study because of their known chemical resistance and use in applications requiring good chemical and aging resistance.

The lining materials were exposed in test cells to 10 actual waste liquids, including two acidic wastes, two alkaline wastes, three oily wastes, a blend of lead wastes, a pesticide waste, and an industrial waste. The polymeric materials were also exposed to three media of known composition, deionized water, 5% aqueous solution of salt, and a saturated solution of low concentration (0.1%) of an organic, tributyl phosphate. The experimental approach and methodology followed are described. The polymeric materials were also exposed to wastes or environmental conditions under a variety of procedures which included primary one-side exposure, immersion-type testing, two types of outdoor exposure, and a pouch test. Some of the exposures were for as long as 2700 days. New methods for the testing of polymeric materials are presented.

The response of the lining material to the waste liquids and test media ranged from little or no effect to severe effects and failure. Oily wastes appeared to have the greatest effect on polymeric membranes. Some organics, even at low concentrations, can have severe effects in long exposures as was shown by the lead waste and the dilute but saturated solution of tributyl phosphate.

Because of the wide differences in the interaction of lining materials and wastes due to their uncertainty and complexity of composition, compatibility testing of the candidate liner and the specific waste or prototype of the waste to be impounded should be part of the process for selecting lining materials for that application.

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FOREWORD

The Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution; it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and provides a most vital communications link between the researcher and the user community.

The following report presents the results of an exploratory experimental research study of the properties and characteristics of a wide range of potential lining materials to control the migration of polluting species from on-land waste storage and disposal facilities into the groundwater. These materials included a compacted native soil, two treated bentonites, hydraulic asphalt concrete, sprayed-on asphalt, and a wide range of polymeric flexible membrane liners that were available during 1975-1980. The effects on the properties of these materials of exposure to a range of real wastes and liquid test media have been assessed in a period of up to seven years.

Francis T. Mayo, Director
Municipal Environmental
Research Laboratory

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Because of the wide differences in the interaction of lining materials and wastes due to their uncertainty and complexity of composition, compatibility testing of the candidate liner and the specific waste or prototype of the waste to be impounded should be part of the process for selecting lining materials for that application.

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ABBREVIATIONS

A - (Soil) activity
°C - Celsius
C - Percent of the clay-size fraction
cm - Centimetre
COD - Chemical oxygen demand
CPE - Chlorinated polyethylene
CR - Chloroprene rubber - neoprene
CSPE - Chlorosulfonated polyethylene
CX - Crystalline or partially crystalline thermoplastic
db - On a dried basis
DMK - Dimethyl ketone - acetone
EPDM - Ethylene propylene rubber
ELPO - Elasticized polyolefin
epi - Ends per inch
EPVC - Elasticized polyvinyl chloride
°F - Fahrenheit
FR - Fabric-reinforced polymeric lining material
GC/MS - Gas chromatography/mass spectroscopy
g - Gram
gpm - Gallons per minute
h - Hours
HAC - Hydraulic asphalt cement
HDPE - High-density polyethylene
i.d. - Interior diameter
IIR - Butyl rubber
in. - Inch
ipm - Inches per minute
K - Coefficient of permeability
L - Liter
LDPE - Low-density polyethylene
LLDPE - Linear low-density polyethylene
LTV - Low-temperature vulcanizing (adhesive)
MEK - Methyl ethyl ketone
mL - Millilitres
MP - Megapoise
MPa - Megapascals
MSW - Municipal solid waste
NBR - Nitrile rubber
P - Poise
P - Pouch number
P_w - (Soil) water content
PB - Polybutylene
PE - Polyethylene
PEL - Polyester elastomer

pen - Penetration
PI - Plasticity index
ppi - Pounds per inch
ppm - Parts per million
psi - Pounds per square inch
PV - Pore volume
PVC - Polyvinyl choride
S-100 - Stress at 100% elongation
S-200 - Stress at 200% elongation
SERL - Sanitary Engineering Research Laboratory University of California, Berkeley, California
R - Fabric-reinforced
TDS - Total dissolved solids
TGA - Thermogravimetric analysis
THF - Tetrahydrofuran
TP - Thermoplastic (or unvulcanized) polymeric lining material
UV - Ultraviolet light
VCM - Vinyl chloride monomer
W - Waste number
XL - Crosslinked or vulcanized polymeric lining material
ρ_β - Soil bulk density
μmho - Micromho

NOMENCLATURE FOR LOCUS OF FAILURE IN ADHESIVE TESTING
AD - Failure within the adhesive
AD-AD - Failure between two coats of adhesive
AD-LS - Failure between adhesive and liner surface
BRK - Break of liner materials outside of seam
CL - Failure at clamp jaw site
DEL - Delamination of the liner material
LS - Failure in the bond between the two adhered liner surfaces

METRIC CONVERSION FACTORS
(U.S. CUSTOMARY UNITS TO SI UNITS)

To convert	Multiply by
Inches to centimetres (cm)	x 2.54
Feet to metres	x 0.3048
Mils to centimetres (cm)	x 2.54×10^{-3}
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Pounds per square inch (psi) to megapascals (MPa)	x 6.895×10^{-3}
Pounds per inch (ppi) to kilonewtons per metre (kN/m)	x 1.751×10^{-1}
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U.S. Customary Units are used in this report as they are commercially used in the United States in the solid wastes industry as well as the liner production and installation industries.

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SECTION 1

INTRODUCTION AND OBJECTIVES

Studies of the land disposal of wastes in the early 1970's clearly indicated the need for positive control measures to prevent contamination of the surface water and groundwater systems that might result from on-land storage and disposal of hazardous wastes. The use of man-made liner materials of low permeability appeared to be a feasible means of preventing pollutants in hazardous wastes from entering the groundwater. At the time this project was initiated in February 1975, a variety of lining materials had been used in various ways for water impoundment and conveyance for 20 years or more. Some of these materials had also been used to line impoundments of brines and some acidic, alkaline, and other waste waters. At that time, a wide range of materials differing in permeability, composition, construction, and cost had been used or were potentially useful for confining possible pollutants. Available information, however, as to the performance and service lives of specific materials exposed to specific wastes was meager. In order to assess the feasibility of using various materials of low permeability to prevent the migration of pollutants into the groundwater for extended periods of time to guide and, perhaps, eventually to regulate the use of lining materials in these applications, the U.S. Environmental Protection Agency (EPA) needed considerably more information.

This project was undertaken in 1975 with the following broad objectives:

- To determine how a selected group of lining materials is affected by exposure to a range of typical hazardous wastes over a relatively extended period of time (initially two years).

- To determine the durability and the cost effectiveness of selected polymeric membranes, sprayed-on membranes, admixed materials, and natural soils as liners for hazardous waste storage and disposal facilities.

- To estimate the effective lives of 12 liner materials exposed to six types of nonradioactive hazardous waste streams under conditions that simulate those encountered in waste impoundment and disposal facilities.

- To develop information needed for selecting specific lining materials for containing hazardous wastes in specific installations.

- To develop methods for assessing the relative merits of the various lining materials for specific applications and for determining their service lives.

- To develop indicator tests that could be used in the selection of lining materials for given applications.

- To assist the EPA in developing effective controls for the proper disposal and management of hazardous wastes.

The concern in 1975 was whether or not lining materials could successfully contain polluting species in hazardous wastes for extended periods. Thus, this project as originally conceived was primarily an exploratory experimental investigation to assess, with respect to typical hazardous wastes, the performance characteristics and durability of lining materials available at the time the work began. These lining materials were either commercially available or, as in the case of the admix and soils materials, prepared from available raw materials. The decision was made in the original design of the project by the EPA to expose these materials to actual waste liquids and slurries removed from storage ponds instead of test solutions or laboratory-manufactured "wastes." It was recognized that actual wastes are, in most cases, complex mixtures of inorganic and organic constitutents generated by more than one source, and their exact composition cannot be determined even though each waste is of a given type. "Typical" wastes of different types were, therefore, selected, characterized, and used in this study.

Since 1975 there have been a number of important developments in liner technology, particularly with respect to polymeric lining materials. Whenever appropriate, these developments have been noted in this report. In addition, some of the newer products were included in some of the secondary exposures, e.g. the pouch and the immersion studies.

Results of the work on this project have been reported over the past several years in the Interim Report on the project (Haxo et al, 1977) at the Second, Fourth, Sixth, Seventh, Eighth, and Ninth EPA Research Symposia (Haxo, 1976, 1978, 1980a, 1981, 1982, and 1983), papers presented at a meeting of the American Society for Testing and Materials (Haxo, 1981), at the Rubber Division, the American Chemical Society, Symposium on the "Role of Rubber in Water Conservation and Pollution Control" (Haxo, 1980b), and at the National Conference on Hazardous and Toxic Waste Management held at the New Jersey Institute of Technology (Haxo, 1980c). In addition, much of this work has been reported in the Technical Resource Documents, Lining of Waste Impoundment and Disposal Facilities (Matrecon, 1980 and 1983).

This final report presents the results of the complete project for the period February, 1975, through July, 1983, and includes information on the exposure of lining materials for periods of up to 2700 days. Most of the exposure data, however, are for periods of up to 1350 days. The reported long-term tests that were initiated early in this project have been continued under EPA Contract No. 68-03-2969. Some of the long-term data obtained in that project are included in this report to present the complete and final results or to present data on which conclusions can be drawn. In many cases, the adverse effects of exposure to the wastes did not become apparent until completion of the longer exposure times.

SECTION 2

SUMMARY AND CONCLUSIONS

SCOPE OF WORK

This exploratory research program was designed to determine the effects of exposing a broad range of selected liner materials to a variety of wastes, sludges, and test media under various test conditions for extended periods of time up to approximately 2700 days. Four broad types of materials were included in the program:

1. One compacted soil of low permeability.

2. Three admixes, consisting of an asphalt concrete, polymer-treated bentonite-sand mixtures, and a hydraulic soil-cement.

3. One sprayed-on membrane of an emulsified asphalt on a nonwoven fabric.

4. Thirty-two different polymeric sheetings based on the following types of polymers:

    - Butyl rubber.

    - Chlorinated polyethylene.

    - Chlorosulfonated polyethylene.

    - Elasticized polyolefin.

    - Neoprene.

    - Polybutylene.

    - Polyester elastomer.

    - Polyethylene (low- and high-density).

    - Polypropylene.

    - Polyvinyl chloride.

Most of the sheetings selected for study in this project were commercial membranes used for lining pits, ponds, and lagoons. Others were prototype materials or sheetings of polymers we felt would be promising as liners. The polymeric materials selected included a range of sheetings of different thicknesses and made by different manufacturers, crosslinked and thermoplastic variations of some of the same polymers, and a few fabric-reinforced sheetings. Some of the commercial materials included samples of sheetings of polybutylene (PB), low-density polyethylene (LDPE), high-density polyethylene (HDPE), and polypropylene (PP) which, though they were not commercial lining materials, were included in the study because of their promising characteristics and their use in the manufacture of pipes and containers for handling corrosive chemicals. These latter sheetings did not contain carbon black nor were they designed specifically for use as linings for waste disposal facilities. Thin films of LDPE and polybutylene had been were used previously in the study of liners for MSW landfills (Haxo et al, 1982).

Note: At the time this project was initiated, lining materials based on HDPE had not been commercially introduced in the United States.

The initially-selected actual wastes representing several general types, included:

- Two acidic wastes, one of which had a pH of less than 1.0.
- Two alkaline wastes, one of which had a pH greater than 12.5.
- One lead waste.
- Three oily wastes.
- One pesticide waste.

These wastes were obtained from ponds in which wastes generated by more than one source were stored. Later an industrial waste containing high concentrations of trace metals and some organics was added.

In addition to the real wastes, three test media of known composition were included for use as reference points:

- Deionized water.
- 5% Solution of salt and water.
- Saturated aqueous solution of tributyl phosphate (0.10%).

The first two media could be used as standard reference points, as many of the wastes that are encountered contain water and salt. The saturated solution of tributyl phosphate was included to assess the long-term effect on polymeric membranes of an organic chemical in a dilute aqueous solution of known low concentration.

Four major types of exposure tests of the lining materials were conducted:

1. One-sided exposure to the wastes. In this exposure test, liner specimens, about 1 ft^2, were mounted in cells and a 1-ft head of waste was placed on them. The polymeric membrane specimens and the sprayed-on asphalt specimens included seams. This test was designed to simulate a liner at the bottom of a pond. The permeability of the material could be assessed by collecting seepage below the liner specimen. Durability was measured by the retention of properties including seam strength after two exposure periods. All four types of lining materials were exposed in this type of exposure test which was the primary exposure test of the study. These exposures consisted of the "primary" exposures of the project and the liners studied were the primary liners.

2. Two-sided exposure to the wastes by immersion of liner slabs. Samples (8 x 8 in.) of 16 polymeric membrane liners were suspended in the tanks of the above cells containing 13 waste liquids and test media. In most cases, two specimens of a membrane were placed in the waste liquid, withdrawn at two different times, and subjected to a series of tests, including measurement of dimensions, weight increase, volatile content, and extractable content, and physical tests such as tensile, tear, and puncture.

3. Outdoor exposure. Two types of outdoor exposure were conducted. The first exposed polymeric membrane samples to weathering, and the second exposed samples intermittently to both weathering and a waste. In the first exposure, three slab specimens of each membrane were mounted on a rack placed on the roof of our laboratory in Oakland, California. One specimen of each membrane was removed and tested after each of three time periods. The specimens were measured for dimensional changes and then tested in a manner similar to those tested in the immersion test. In the second type of outdoor exposure, sheetings of polymeric membranes were used to line small tubs which were then loaded with wastes. This test allowed assessment of the effects of weather and waste exposure on the liner below, above, and at the interface of the waste and air; it also assessed the effect of directional orientation with respect to the sun. The liner specimens in the tubs contained a "field" seam so that the effect of wastes on seam strength could also be assessed.

4. A pouch test. This test furnished two types of information regarding a membrane liner:

    1. Information regarding the permeability of the membrane to waste constituents.

    2. The effect of one-sided exposure of the membrane to a waste.

In this test pouches were fabricated from polymeric membranes filled with wastes and sealed. The sealed pouches were then immersed in deionized water and exposed for extended periods. The permeability of the membranes could be assessed by measuring at various time intervals the weight change of the pouch and the conductivity and pH changes of the deionized water in which the pouches were placed. When the pouches were eventually dismantled, dissected, and tested, they also yielded information on one-sided exposure to wastes.

The duration of these tests ranged up to 2700 days for the primary tests, 1456 days for the immersion test, 1231 days for the roof rack test, and more than 2500 days for some of the tubs. Some of the pouch tests ran as long as 2000 days.

The project was concerned largely with the chemical compatibility of liner materials as measured by the liner's absorption of the waste solution, by changes in mechanical properties and, in some cases, by changes in permeability.

> Note: Chemical compatibility of semicrystalline polymeric materials with various wastes, as measured by environmental stress-cracking, was not addressed in this project. These materials were not significant factors in the USA at the initiation of this project. Studies of the environmental stress-cracking resistance of semicrystalline materials under different conditions are underway in a current project by Matrecon (EPA Contract No. 68-03-3169).

In addition to the exposure studies, a variety of analytical tests were developed for characterizing and fingerprinting the polymeric membranes. These included analyses for ash, volatiles and extractables, and a pyrolysis test to measure the general composition.

The overall results of the project indicated that the response of the lining materials to the wastes varied considerably, from apparent compatibility to obvious failure. Because liner-waste combinations are highly specific, compatibility testing is needed as part of the process to select a liner for a given waste.

Specific conclusions of this work from the viewpoints of lining materials, wastes, and test methods are presented below.

Lining Materials

Native soil--

Specimens of the native soil (Mare Island) liner showed only modest effects during the 6.5 years of exposure to five of the waste liquids:

- Low alkalinity waste, "Spent caustic."

- Lead waste.

- Oily wastes:
  "Slurry oil"
  "Oil Pond 104."

- Pesticide waste.

Combinations of the soil liner with the acidic wastes had been eliminated in the screening tests because of apparent incompatibility, as evidenced by early passage of low pH liquid through a 2-in. core.

The 1-ft thick soil liners showed initial low permeability (2.0-3.1 x 10^{-8} cm s^{-1}) to all the wastes tested, which they maintained throughout the exposures. The apparent insensitivity of this soil to various chemicals is probably related to its high ratio of nonclay to clay minerals in the fraction smaller than 2 µm. Another feature of this soil that may contribute to its insensitivity was its high salt content; it had been dredged from the channels in the Carquinez Straits in California at the mouth of the Sacramento River in San Francisco Bay and had dried on land. Except for the possible migration of copper, the other five metals tested (cadmium, chromium, lead, mercury, and nickel) did not appear to migrate below about the top 2 cm of the liner during 958 days of exposure to the "Oil Pond 104" waste which contained these trace metals. A liner of an equivalent initial permeability to water and with high amounts of smectite may have given different results.

Asphalt Cement--

Combinations of the asphalt concrete and the oily wastes were eliminated in the screening tests. However, in spite of the low permeability and good mechanical properties of the 2.5-in.-thick asphalt concrete, the liner was deficient in several other exposures. Both specimens in contact with the strong acid ("HNO_3-HF-HOAc") developed leaks; some of the aggregate at the surface was dissolved, and the asphalt itself hardened severely during exposures that were relatively short (40 and 199 days). Leaks also developed in the specimens below the "Spent caustic" and lead wastes. The lead waste contained sufficient oily constituents to cause the asphalt concrete to become almost a slush. Some seepage also occurred through the specimens below the liner. We conclude that this type of lining material should not be used with wastes that contain oily compounds and that the aggregate proposed for use needs to be thoroughly tested for compatibility with the waste. Also, a thickness of 2.5 in. may be insufficient even for water and compatible dilute wastes.

Bentonite and Sand Mixtures--

Polymer-treated bentonite and sand mixtures were eliminated in the screening test from exposure testing with the acid and strong alkaline wastes but were tested in cells containing the lead, pesticide, "Slurry oil," and "Oil Pond 104" wastes. The 5-in. thick specimens exhibited low permeability ($<10^{-8}$ cm s^{-1}), though the Bentonite B specimens showed significantly lower permeability than did the Bentonite A specimens. This type of lining material is probably not satisfactory for oily type wastes since considerable "fingering" of the waste into the liner mass took place during the 980 days of exposure; however, none of the oil broke through the liner to be collected in the base. Even though some flow through the liner occurred, the distribution of the metals at the various depths was uniform. The use of a soil cover on the liner to produce an overburden could probably reduce the fingering effect and reduce flow.

Soil-Cement--

The 4-in. thick soil-cement specimens showed resistance to "Spent caustic," the lead waste, the two oily wastes, and the pesticide waste. Soil-cement had been eliminated from exposure to the strong acid waste ("HNO_3-HF-HOAc") in the screening test. No seepage occurred in any of the specimens. In the five recovered and tested, actual increases in compressive strength occurred. Metal concentrations were uniform through the depth of the liners.

> Note: It must be recognized that these specimens were all small and not subject to shrinkage or cracking that would be experienced in large installations.

Sprayed-on Asphalt--

When exposed to the "Spent caustic" waste, the pesticide waste, and water, the sprayed-on asphalt showed little change in properties. A leak developed in one of the specimens in a cell containing brine. The sprayed-on asphalt had been eliminated from exposure testing to the oily wastes because of their oily contents, and screening tests showed it to be incompatible with the highly acidic waste ("HNO_3-HF-HOAc"). In contact with the lead waste, it softened considerably and in all cases absorbed water. Compatibility tests would be appropriate when considering sprayed-on asphalt for lining an impoundment, and all oily or highly acidic wastes should be avoided.

Polymeric Membranes--

There were major variations in the responses of the polymeric membranes to individual wastes, particularly to those wastes containing oily constituents. The effects varied from essentially no change during the exposures to complete failure. Several of the membranes were deleted in the screening tests from exposure to the oily wastes in the primary cells. The variations in responses were not only among the different polymer types, but also within a given polymeric type due to compound variations, e.g. plasticizer type and amount, crosslinking, and fabric reinforcement. These results demonstrate the need in the selection and design process for determining the compatibility of the individual lining material with the particular waste with which it is to be in contact.

The general types of membrane liners discussed below include crosslinked polymers, thermoplastic polymers, semicrystalline polymers, and fabric-reinforced membranes with coatings based on either crosslinked or thermoplastic polymers.

Crosslinked polymers--This group of polymeric membranes includes:

- Two based upon butyl rubber.
- Two based on ethylene propylene rubbers.
- Three based on neoprenes.
- One based on chorinated polyethylene.

The membranes include both fabric-reinforced and unreinforced sheetings which were tested in both the primary and immersion tests.

Crosslinking normally causes a polymer to swell less and be more resistant to change in liquids. This quality was observed in the lining materials based on chlorinated polyethylene (CPE) which, in all but one case, experienced less swelling and fewer changes in properties with the crosslinked materials. In contrast, a thermoplastic ethylene propylene rubber (EPDM) sheeting had significantly lower swelling in the wastes than a crosslinked EPDM, which is the usual type of EPDM sheeting used for liners. It must be recognized, however, that there are considerable differences in the EPDM rubbers, some of which can contain some crystallinty, which tends to reduce swelling.

The seams of the butyl rubber, EPDM and neoprene were all prepared with cold-curing adhesives which tended to yield low values. In tests the failures were in the adhesives.

Thermoplastic polymers--The thermoplastic sheetings selected for this project included:

- Two based on chlorinated polyethylenes.
- Four based on chlorosulfonated polyethylenes.
- Eight based on polyvinyl chlorides.
- One based on an ethylene propylene rubber.

All of these materials tended to be sensitive to the type of waste, indicating the need for compatibility testing before selected for use as a liner in a waste storage or disposal facility.

Membranes based on polyvinyl chloride (PVC) varied considerably from sample to sample and in their responses to a given waste liquid. Test results for the specific materials tested varied from major losses in weight to significant increases, and from near embrittlement to severe softening. This variation indicated the effect of compound differences, e.g. plasticizers. The need for compatibility testing of the liner with the waste was quite apparent.

The seams of the membranes based on thermoplastics retained their strengths during most of the exposures.

Semicrystalline polymers--A variety of semicrystalline materials were exposed in this project, including:

- One elasticized polyolefin.
- One high-density polyethylene.
- Two low-density polyethylenes.
- One polybutylene.
- One polyester elastomer.
- One polypropylene.

Of these semicrystalline polymers, only the elasticized polyolefin (ELPO) was a commercial product for liner use at the time this project was initiated. The polyester elastomer (PEL) was an experimental material. The remainder were all commercially-manufactured films, but were not for liner use. All of the latter were clear and contained no carbon black. They were tested because their good chemical resistance indicated potential use in liner materials.

Among the polymeric materials studied in this project, the sheetings of semicrystalline materials showed the least absorption of wastes and, by and large, retained their physical properties best. Variations arose in that some of the crystalline materials, particularly the elasticized polyolefin, swelled considerably in wastes that contained oily constituents. The elasticized polyolefin had the lowest level of crystallinity on the polymers in the above group.

The heat-sealed seams in the ELPO and polyester elastomer specimens essentially maintained their strength during the exposures; for both specimens, the lower values after exposure reflected softening of the liner itself.

The polyester elastomer sheeting exposed in the strong acid ("HNO_3-HF-HOAc") failed completely and cracked. The ELPO absorbed considerable amounts of the alkaline "Slop water" waste and showed a major increase in permeability after about one year in the pouch test.

Note: Since this project was initiated, new crystalline polymeric sheetings based on several of these polymers have been developed for use as lining materials and have become commercially available.

Wastes

In terms of their effects on the lining materials, the 13 wastes and test media used in the project can be grouped as follows:

- Acidic wastes.
- Alkaline wastes.
- Brine and industrial wastes.
- Lead waste.
- Oily wastes.
- Trace organic test liquid.
- Deionized water and pesticide waste.

Overall, the deionized water had the least effect on the membrane liners except for those based on elastomeric polymers containing chlorine; in the case of the sheetings based on neoprene, CPE, and CSPE the swell was greater than in a waste containing salts.

Among the wastes included in the program, those with oily constituents generally caused the greatest swelling and the greatest loss in the values of the properties of the polymeric and asphaltic lining materials. These wastes included the lead waste, the "Slurry oil," "Oil Pond 104," and "Weed oil."

Two types of nonoily wastes, i.e. acidic and alkaline, caused significant loss of plasticizer and stiffening of the PVC specimens.

A trace (<0.1%) of an organic species such as tributyl phosphate in an aqueous solution caused severe swelling and loss of physical properties of some membrane liners after long exposure. The effect of immersion in the dilute, but saturated, tributyl phosphate solution varied considerably among the 16 membranes, indicating a specificity of liners and organic constituents. Semicrystalline materials showed the lowest effects of the trace organics in the wastes.

The nonhomogeneity of wastes and the sampling procedure posed problems, and could pose major problems, in testing the compatibility of membranes and wastes in making the selection of a lining material for impounding a given waste. Some of the wastes stratified so that the waste at the bottom of a cell or tank had a considerably different composition from that at the top. Thus the specimens exposed horizontally at the bottom of a cell were not exposed to the same waste that suspended specimens were exposed to at the top. Such could also be the case with a pond liner when exposure at the bottom is compared with exposure near the top. Thus the possibility exists that a test specimen in a compatibility test may not be exposed to a representative waste from the impoundment or to a waste from a critical area in the pond. Two of the wastes were saturated with salts that crystallized out of the waste onto the liner, thus exposing the liners to wastes different from the initial waste solutions. Constant agitation of a nonhomogeneous waste during exposure testing may be a solution, but it may not result in the worst exposure condition. The effect of agitation should be studied in order to establish a compatibility test. More laboratory data are needed to develop background correlations with field exposure and actual service.

Within their respective groups, the more acidic and the more alkaline the wastes, the more aggressive to the lining materials. The "Spent caustic" waste, which is essentially a saturated brine solution, behaved very much like the 5% brine and the industrial waste.

Test Methods

The various test methods that were used in characterizing the liners and following changes on exposure to waste liquids revealed significantly different responses among the liners on exposure to each type of waste. Generally, some correlations appear to exist among the results obtained by the different test methods.

Primary Exposure Tests--

The primary exposure cells and the test procedures were satisfactory for assessing the effects of the waste exposure on the liners. These were one-sided exposures. The cells acted as permeameters with a head of 1-ft of waste liquid and yielded permeability data on the soil and bentonite compositions. The high acidic wastes, however, resulted in corrosion of the steel containers, presumably through pinholes in the epoxy coating. At the time the cells were dismantled, those cells that had contained acidic wastes were so badly damaged that they could not be reconditioned. The anti-rust paint used on the outside of the cells generally was not satisfactory for the longer exposure periods; the cells had to be repainted during service. In more recent projects, at the time the cells were fabricated, they were epoxy coated on the outside as well as on the inside.

Test duration should be as long as possible. The 12 and 24-month exposures originally set in the contract were far too short for estimating service lives unless the latter are relatively short. Also, the number of exposure periods was inadequate for projecting some of the physical properties because of inherent errors arising from the nature of these tests and the problems in handling exposed specimens.

Immersion Test--

The immersion test in which both sides of the membrane were exposed to waste yielded somewhat faster responses. The test also allowed us to greatly increase the number of membranes that were exposed.

An immersion test can be adapted for use as a compatibility test of a lining material with a given waste if at least four time exposures are used with at least one month between exposures. However, the immersion test should be investigated further to develop correlation with actual experience.

Roof Rack Exposure--

The roof rack test as run in this project was an exploratory test to assess the effect of weather exposure on a selected group of membrane liners. Conditions of exposure in Oakland, California, are comparatively mild; however, the exposure period of three and one-third years showed many of the effects that are encountered in actual exposures of liners in service. Most of the liner materials are at times exposed to the weather, although several are generally covered with soil in actual service.

All of the membranes showed some changes in properties but none of the liners had actually aged to the point at which their properties had seriously deteriorated. Most of the membranes lost in elongation at break and in weight and extractable content due to evaporation of plasticizers; they all shrank and tended to stiffen, but generally gained in tensile strength. The CSPE membrane gained some weight. The PVC liners showed surface crazing and were the most severely weathered, as was expected; in actual service, the PVC liners are generally soil covered.

Tub Test --

The tub test demonstrates that different locations within an impoundment can have significantly different effects on the retention of properties by a liner. This test also shows that the most severe effects of exposure occur at the interface between the waste and the weather.

Pouch Test --

The pouch test used in this project with only thermoplastic and crystalline membranes shows considerable promise for assessing the effects of a waste liquid in contact with a polymeric membrane liner. Efforts should be undertaken to increase the applicability of this type of test to crosslinked sheeting and to stiff, thick sheetings. The test was run primarily with thinner sheetings that could be heat-sealed or solvent-seamed. The pouch tests, however, may have to be run for extended periods of time to observe long-term effects, and they may require interpretation until this test procedure has been used extensively.

Seam Strength Tests--

The changes in strength of the seams incorporated in the primary exposure specimens were tested in both peel and shear modes. The effect of the exposure on the seam itself is shown best by the peel test; effect of the change in the shear values generally relate to the effects of the wastes on the liner itself. We recommend that both tests be performed on the seams.

Environment Stress-Cracking Resistance--

Note: As high-density polyethylene has become an important material for the manufacture of liners, accelerated environmental stress-cracking tests should be developed to test the compatibility of crystalline polymeric materials with wastes. The resistance to environmental stress-cracking among semicrystalline materials is presently tested by immersions in standard surfactant solutions. The possible stress-cracking in various waste liquids needs exploration. These effects are not applicable to elastomeric and noncrystalline thermoplastic and were not studied in this project.

SECTION 3

TECHNICAL APPROACH AND RESEARCH PLAN

BASIC INFORMATION REGARDING LINERS NEEDED BY EPA AND THE WASTE MANAGEMENT COMMUNITY

The U.S. Environmental Protection Agency, in their report to Congress (EPA, 1974), concluded that the then existing current management of the nation's hazardous wastes was "generally inadequate" and that the "public health and welfare are being unnecessarily threatened by the uncontrolled discharge of such waste materials into the environment," and recommended control of the disposal of these wastes. In response, Congress passed the Resource and Recovery Act of 1976. To implement this law and develop the necessary controls and regulations, the EPA needed technical data to establish the types and properties of barrier materials that could be used to prevent migration of potentially polluting species in a waste storage or disposal facility from entering and contaminating surface water and groundwater. From a broader viewpoint, the EPA needed to establish the state of liner technology and to determine whether the technology existed for adequate on-land storage and disposal of wastes. They needed to establish the limitations of the then-current technology and demarcate the problem areas and the areas of uncertainty toward which future research should be directed.

At the time this project was initiated (1975), information regarding the effectiveness, compatibility, and durability of lining materials in contact with the wide range of hazardous and industrial wastes was very limited. Man-made materials of low permeability had been used to line water impoundments and canals for a considerable length of time and much information regarding these materials had been developed. Requirements for resistance to the permeation of water in these applications were not stringent as minor losses of water can be tolerated. Also, the interactions of water with many of these materials were well known. However, in the case of hazardous wastes, the situation is drastically different. Such wastes contain a vast array of organic and inorganic constituents, many of which are toxic; therefore, the tolerance for permeation of waste constituents from land impoundments is necessarily very low because of potential contamination and pollution of surface water and groundwater.

A lining material for a waste storage or disposal facility must have low permeability and maintain its integrity for the time period required. It should not change in properties; particularly, it should not increase in permeability. Also, a liner should probably be capable of deforming and accepting small deformations of the surface on which it is placed.

Furthermore, information was needed by designers and facility owners to aid in the selection of materials that could be used in the lining of disposal facilities. It was desirable while conducting the project to develop information that would help designers understand the various liner materials and their properties.

EXPERIMENTAL APPROACH

To meet these broad needs for information our basic approach was primarily exploratory in nature. As a first step we wanted to extend the information which we had accumulated in the project relating to liners for containing municipal solid waste (Haxo et al, 1982). In the two years after the municipal solid waste project had been initiated in 1973, liner technology had progressed and several new lining materials had been introduced. We needed to take advantage of these newer materials so that we could establish the state of current technology. On the other hand, in order to develop a depth of information on some of the most-used materials, it was desired to include in this study some of the same membranes that had been put into exposure with MSW leachate.

The lining materials that were to be investigated were primarily commercial products. In the case of field-prepared lining materials such as soils and admixes, standard methods and materials were to be used in their preparation as test specimens. One or two compacted native soils were to be included in this set of lining materials. All of the liners in the exposure studies were to be highly characterized so that information on them could be transferred to other lining materials of somewhat different composition and construction.

It was decided from the outset to use real wastes in the exposure tests. These real wastes were to be typical of the wastes that might be encountered and might be stored or disposed of on land. The wastes selected and used were also to be highly characterized particularly with respect to constituents that might be aggressive to lining materials. Again, such information would aid in developing an understanding of the effects of wastes on lining materials and could be used in assessing the aggressiveness of other wastes. The initial program called for six wastes, but later this number was increased to ten. It was particularly important to include highly acidic and highly alkaline wastes, and wastes that contained organic constituents. Consideration of these characteristics contributed to the selection of the specific wastes that were used.

It was recognized from basic knowledge of materials and past experience that some combinations of liners and wastes would be incompatible and could lead to early failures in the test cells. It was desirable to avoid liner-waste combinations that would probably fail after relatively short exposures so that maximum use of the equipment and the funds available could be made. Thus, we selected specific lining materials for exposure to each waste so that as many combinations as possible could be tested for the full, predetermined exposure periods. A series of short, preliminary compatibility tests of candidate lining materials and the wastes were performed to screen out the obviously incompatible combinations.

Exposure conditions should simulate the environment and other aspects of actual service. To estimate the durability or the service life of a lining material, the environmental conditions to which it is exposed must be known and understood. The service conditions encountered by a liner for a hazardous waste impoundment contrast greatly with those encountered by a liner in a landfill. In the latter case, the usual environment of a liner is anaerobic, cool, dark, and continually moist (Haxo, 1976). Also, the waste fluid or leachate, though variable from landfill to landfill and even within a landfill, will not be as variable, or as highly concentrated in pollutants or in constituents that are aggressive to liner materials, as the waste liquids encountered in a waste disposal facility that contains industrial, hazardous, and toxic wastes.

Some of the conditions that can be encountered by liners in hazardous waste impoundments are:

- Exposure to a vast array of different chemical species in direct contact or under soil cover.
- Exposure to weather, e.g. sunlight, rain, wind, ozone, and heat.
- Wave action of the liquid waste in the pond.
- Intermittent exposure to both waste liquids and weather.
- Low and high temperatures.
- Burrowing and hoofed animals.
- Ground movement.
- Irregularity of the soil above and beneath a liner.
- Changing temperatures during the day.

These conditions must be recognized in evaluating materials in the laboratory or on a pilot scale.

The durability of the lining materials was assessed by measuring the properties after two exposure periods. Each type of material has its own characteristics which can be used to follow changes with exposure time, although in all cases low permeability to waste constituents is an essential characteristic that a liner must maintain during exposure to a waste. Major changes in these characteristics are indicative of lack of compatibility of a liner material with the waste liquid.

As originally planned, the two exposures were 12 and 24 months; however, it became apparent by the small changes in properties in the early stages of the project that longer exposure periods would greatly increase the value of the test results.

RESEARCH PLAN

The basic research plan, with modifications during the course of the project, consisted of the following:

- To contact the manufacturers of polymeric lining materials and other organizations involved in liner fabrication and installation regarding the current status of liner technology, and requesting most recent information on their respective products and their performance characteristics. This aspect of the plan was continued throughout the entire project as liner technology developed rapidly during this time with the result that new materials were being offered and several materials were dropped from the market.
- To design and construct primary cells which simulated the environment of a liner in service at the bottom of an impoundment. The liner specimen had to be of sufficient size to allow for the measurement of several physical properties. Initially it was proposed that the materials under test should line the entire interior of the primary cells. However, this was not possible with the soil, admix, and asphaltic lining materials, as it was decided that all materials should be treated equally. The cells were designed so that the liner specimens would lie flat underneath a 1-ft head of waste so that, if waste permeated through a liner specimen, it could be collected. Specimens of the polymeric membranes were designed to incorporate a field-type seam.
- To select, collect, and analyze real wastes typical of a variety of wastes that would commonly be encountered in practice. These included acidic, alkaline, lead, oily, and pesticide wastes.
- To obtain, test, and select polymeric membrane lining materials for the primary exposure test. Bases for selection: if possible, a range of polymeric types, one thickness, i.e. 30 mil, unreinforced (no fabric) membrane, and some of the same membranes used in the study of lining materials for MSW landfills. The remaining membranes were to be tested in immersion and other laboratory exposure tests.
- To obtain candidate soils, aggregates, and other raw materials for testing in soil and admix compositions. Once the soils were obtained, to optimize compaction and design for low permeability and durability to water and wastes and to make the final selection of materials and design on the basis of these characteristics.
- To prepare the liner specimens, mount them in cells, and load the cells with the appropriate wastes. Part of the preparation of the polymeric membrane specimimens included the incorporation of field-type seams.
- To monitor the test cells and to collect, quantify, and characterize the seepage.

- To remove the specimens from the cells at appropriate time intervals and to measure the properties so as to determine the magnitude of change during exposure (from the original values for the respective properties).

- To expand the matrix of liner materials and wastes by performing immersion-type tests in which smaller test specimens would be hung in the tanks above the primary liners.

- To assess the outdoor weathering of selected membranes, both on a roof rack and as linings of small tubs that contained different wastes.

- To develop methods of characterizing liner materials and of testing their properties and performance.

- To assess the permeability of different polymeric membranes to different components of wastes by a pouch test in which pouches of membrane liners could be filled with a waste, sealed, and placed in deionized water. The weight of the pouch and the electrical conductivity and pH of the deionized water would be monitored to assess the permeability of water into the pouch and ionic and organic material into the water.

SECTION 4

SELECTION AND CHARACTERIZATION OF WASTES AND SLUDGES

INTRODUCTION

In designing the original program, EPA desired that real wastes be used and that as broad a range of types as possible be included. The EPA project officer identified seven basic types of wastes which should be included. These were:

1. A strong acidic waste.
2. A strong alkaline waste.
3. A lead waste from gasoline tanks.
4. An oil refinery tank bottom waste.
5. A pesticide sludge.
6. A saturated hydrocarbon sludge.
7. An unsaturated hydrocarbon sludge.

SELECTION OF SPECIFIC WASTES

Twelve individual sludges and wastes were collected by Industrial Tank, Martinez, California, and a preliminary characterization was made of one-gallon samples of each in order to select the specific wastes to use. Eleven of the twelve samples met the basic requirements of composition for this project. Two hundred gallons of an individual waste were required for the exposure testing, but several were not collected in that amount. For example, it was necessary to combine the three lead wastes. Another waste, that had been labeled "aromatic oil waste," contained little oil and was volatile; it was discarded. Subsequently, a heavy aromatic oil was obtained from Cities Service.

In several cases the samples that arrived in drums, as well as the corresponding one-gallon samples, were highly inhomogeneous. Furthermore, in preliminary analyses, samples from the two sets were found to be considerably different. These differences affected the selection of the materials, the handling of the waste, and the actual exposure testing of the liners. Wastes that contained water, oils, and solids stratified. Generally, the solids fell to the bottom and lay on the liner and the oil rose to the top and did not directly contact some of the liners, although some dissolved and emulsified oils did contact the liner. The inhomogeneity of the wastes pointed to a basic problem in the testing of liners with a given waste because it showed that the liner could be in contact with several different materials. Test results also showed the importance of proper sampling.

The specific wastes that were included in the exposure testing in this project are listed in Table 1.

TABLE 1. WASTE LIQUIDS USED IN IMMERSION STUDY OF LINERS

Serial number	Waste description	
	Type	Identification
2	Alkaline	"Spent caustic"
4	Alkaline	"Slop water"
5	Oily	"Oil Pond 104"
7	Oily	"Weed oil"
9	Acidic	"HNO_3-HF-HOAc"
10	Acidic	"HFL"
11	Pesticide	"Weed killer" waste
14	Lead	Lead waste blend
15	Oily	"Slurry oil"
16	Industrial	"Basin F" water
17	...	"Well 118" water
18	...	Deionized water
19	Brine	5% NaCl solution
20	Trace organic	Water saturated with tributyl phosphate (0.1% conc.)
21	...	50:50 blend of Well 118 water and deionized water

Description of the Wastes in the Test Program

Acidic Waste--

Two acid wastes were received from Industrial Tank. The first was labeled, "HNO_3-HF-HOAc," and the second, "HFL." Four 55-gallon drums of the first waste were received. This was a mixed acid containing predominantly (16%) nitric acid, but also included some hydrofluoric acid and acetic acid. It was predominantly aqueous, was straw colored, and had a trace of suspended solids. The viscosity of this waste was essentially that of water.

This acidic waste was used in all types of the exposure testing.

Only one drum of the second waste, "HFL," was received. It also was an aqueous solution. It was used in the latter stages of the project in immersion tests and in limited exposure testing in the primary cells.

Alkaline Waste--

Two alkaline wastes, labeled "Spent caustic" and "Slop water," were received from Industrial Tank.

"Spent caustic" waste--Four drums or approximately 200 gallons of spent caustic was received. It was essentially an aqueous solution, tea colored, and had crystals in suspension. This waste was saturated and, when cold, much salt precipitated. The solution had a solids content of above 20%. It caused considerable problems in the pumping due to the crystal formation which damaged the centrifugal pumps.

This waste was used in the primary exposure cells, in immersion tests, and in some of the pouch tests.

"Slop water" waste--Only one drum of this waste was received. It also was an aqueous solution with a high salt concentration and a somewhat higher pH than the spent caustic. It was used in immersion and pouch tests and in a limited number of cells.

Lead Waste From Gasoline Storage Tanks--

Three different lead wastes (identified as W-3, W-8, and W-12) were initially received from Industrial Tank. In order to have sufficient lead waste for the primary exposure test, the three were combined to yield four drums of waste and assigned the Matrecon serial number W-14. This combined waste had a strong gasoline odor; it contained some low boiling fractions, lead compounds, and other additives. However, it was basically an aqueous waste. These wastes were obtained in the washing of tanks containing leaded gasolines.

This waste was used in the primary exposure cells, in immersion tests, and in some of the pouch tests.

Oily Wastes--

Several oily wastes of substantially different character were obtained and included both unsaturated and saturated oils. These included wastes labeled "Oil Pond 104," "Slurry oil," and "Weed oil."

"Oil Pond 104" waste--Five drums of waste from "Oil Pond 104" were received and appeared to contain dispersed water. However, all of the tests run on this waste showed it contained oily material and solids. The oil phase contained both saturated and unsaturated oils. It was a relatively viscous oil used as a road oil which required some heating in order to be handled and poured.

The "Oil Pond 104" waste was used in the primary exposure tests and in the immersion tests.

"Slurry oil" waste--One hundred gallons of "Slurry oil" waste was received from Cities Service in Louisiana. It was a heavy highly condensed hydrocarbon and contained several percent of suspended solids believed to be residual catalysts. For this test, it was considered to be an "Aromatic oil" waste. It was necessary to heat this oil in order to pump it into the cells. Because of the lesser quantity of oil, its use in the primary cells was limited.

Four drums of another oily waste were received from Industrial Tank which were labeled "Aromatic oil" waste. However, the oil content of this waste was small and highly volatile. Consequently, it was not used in any of the tests because, in the loosely covered cells that were used, the oily component would have evaporated and there was insufficient waste to replenish the cells.

"Weed oil" waste--One drum of "Weed oil" waste was received from Industrial Tank. It was predominantly an aqueous waste that contained low molecular weight hydrocarbons which appeared, by subsequent tests, to have been highly aromatic. The oil fraction had been used as a "Weed killer" for spraying between the ties on railroad tracks.

The "Weed oil" waste was used in immersion tests, in pouch tests, and in a limited number of cells.

Pesticide Waste--

Five drums of a waste labeled "Weed killer" were received from Industrial Tank. This waste was primarily a wash water which contained some suspended clays and a herbicide. The solids settled out rather rapidly after stirring.

This waste was used in the primary cells and in the immersion tests.

Additional Wastes and Test Media--

In addition to the wastes discussed above, which were used in the major exposure of this project, several additional wastes and test media were added to the project, primarily for use in immersion tests. Some exposure cells were filled with these wastes as a part of the immersion tests. Consequently, some of the liners were exposed to them as primary liners. These wastes included:

- An industrial waste, labeled "Basin F," which contained both metal and organic constituents.

- A contaminated water, labeled "Well 118."
- Deionized water.
- A brine consisting of a 5% aqueous solution of NaCl.
- A saturated solution of tributyl phosphate in distilled water.

The last three media are controlled reference compositions. Deionized water was included to assess the exposure by the different liner materials in a medium of zero concentration of the different dissolved constituents which would allow estimation of the effects of concentrations. The 5% NaCl brine has a moderate salt content in the range of municipal solid waste leachate; it is a reference material used in the immersion testing of polymeric materials and has moderate concentrations of ions. Also, we used it in pouch testing of membranes so that the ions could be traced. The last immersion medium was incorporated into the test program to assess the effects of trace amounts of organics in solution; tributyl phosphate which has a slight solubility in water (0.1% at 23°C) was used for this purposes.

ANALYSIS AND CHARACTERIZATION OF THE WASTES

Inasmuch as the wastes being used were real wastes, which were not only multiphase but also highly complex from the standpoint of composition, it was necessary to perform relatively intensive analyses in order that the components in the wastes that were aggressive toward different liners could be identified. It was particularly important to know as much as possible about the quality of the waste in contact with the liner under test. During the course of the project, these wastes were subjected to several analyses to develop this information. Initial analyses were performed by the Sanitary Engineering Research Laboratory (SERL) of the University of California, Berkeley. At that time no consistent set of procedures had been established to analyze these wastes. The initial analyses did not recognize the multiphase character of the wastes as they were treated as single materials. Following the description of the analyses used by Dr. Robert Stephens (1976) of California Health Services, our analytical procedure was modified to follow a procedure in which the waste was separated by either centrifugation or other appropriate method into:

- An organic liquid phase containing dissolved organics and dissolved water.
- An aqueous phase containing dissolved organics and dissolved inorganics.
- A solids phase which can be inorganic or organic solids.

Details of the analytical procedure are described in Appendix B and results of the analyses are presented in Table 2. The analyses are based upon samples of the blends of the wastes as they were loaded into the cells. The procedure for the blending from the several drums is described in the next section.

TABLE 2. ANALYSIS OF HAZARDOUS WASTES USED IN PROJECT

	Wastes[a]								
	Acidic		Alkaline			Oily waste			Pesticide
Phases and tests	"HFL" (W-10)	"HNO_3-HF-HOAc"[b] (W-9)	"Slop-water" (W-4)	"Spent caustic"[b] (W-2)	"Lead Waste"[b] (W-14)	"Slurry oil" (W-15)	"Oil Pond 104"[b] (W-5)	"Weed oil" (W-7)	"Weed killer"[b] (W-11)
Separation of phases									
Phase I, aqueous insoluble organic liquid, weight %	0	0	0	0	5.0	98	89	20.6	0
Phase II, aqueous phase, weight %	100	100	100	95.1	86.2	0	0	78.4	99.5
Phase III, solid phase, weight %	0	0	0	4.9	3.4	2.0	11	0	0.5
Phase I - Organic									
Weight %	...	0	...	0	10.4	98	89	20.6	0
Flash point[c], °C	...	...	...	...	<20	174	157	...	...
Viscosity[d], cP									
At 20°C	...	...	...	...	...	3200	300	...	...
At 30°C	...	...	...	...	...	660	124	...	...
Water content, %	...	...	...	...	...	0	17	...	...
Organic group[e]									
Asphaltenes, %	...	...	...	...	...	13.1	9.6	...	...
Polar compounds, %	...	...	...	...	...	14.0	18.6	...	...
Saturated hydrocarbons, %	...	...	...	...	...	13.1	37.9	...	...
Aromatics, %	...	...	...	...	...	59.7	339	...	...
Lead, mg/L	...	...	...	...	530	...	170[f]	...	...
Phase II - Aqueous									
pH	4.8	1.5	12.0	11.3	7.6	...	...	7.5	3.1
Weight %	...	100	...	95.1	86.2	0	0	78.4	99.5
Solids in solution, %									
Total	2.48	0.77	22.43	22.07	0.9	...	36	1.81	0.78
Volatile	0.9	0.12	5.09	1.61	0.35	...	31	1.0	0.46
Solids, total g/L	...	140	...	234.5	3.23	...	...	9.10	6.78
Volatiles, g/L	...	15	...	24.2	1.62	...	...	3.45	3.32
Total dissolved, g/L	...	137	...	234.5	2.66	...	...	1.75	6.62
Volatiles dissolved, g/L	...	7.0	...	24.0	1.14	...	...	...	3.22
Total suspended, g/L	...	15.0	...	0.04	0.41	...	...	...	0.16
Volatiles suspended, g/L	...	9.0	...	0.01	0.28	...	...	...	0.10
Alkalinity, g $CaCO_3$/L	...	...	..	8.69	1.06/28	...	...	...	25
Oil and grease g/L	...	0.0	...	0.02	0.15	...	...	...	0.05
Soluble volatile organics, mL/L	...	0.0	...	0.15	1.0	...	...	...	0.8
Lead, mg/L	...	28[f]	...	5.0[f]	13	...	...	...	1.4[f]
Phase III - Solids									
Weight %	...	0	...	4.9	3.4	2.0	11.0	...	0.5
Flammability									
Flame	...	...	...	Yes	...	...	Yes	...	Yes
Color	...	...	...	Orange	...	...	...	...	Orange
Smoke	...	...	...	No	...	...	...	...	No
Solids, %									
Organic,	...	...	...	8.9	22.5	...	78.9	...	50.4
Inorganic, %	...	...	...	91.1	77.5	...	21.1	...	49.6
Water extract, mg/g	...	...	...	122.4	43.8	...	11.2	...	3.5
pH	...	...	...	5.2	7.4	...	8.4	...	2.5

[a] Matrecon waste serial number shown below identification.

[b] Analyzed after exposure.

[c] ASTM D92.

[d] ASTM D2983.

[e] ASTM D2007-69, in percent by weignt.

[f] Total lead content of the waste.

These results, however, did not identify specific constituents materials in the wastes. Consequently, five of the real wastes were subjected to a more intensive analysis which included gas chromatography/mass spectroscopy (GC/MS). These analyses were performed by EAL Corporation of Richmond, California (Appendix C). The wastes analyzed were:

- Acidic waste ("HNO_3-HF-HOAc"), W-9.
- Alkaline waste ("Spent caustic"), W-2.
- Lead waste blend, W-14.
- "Oil Pond 104" waste, W-5.
- Pesticide waste ("Weed killer"), W-11.

In these analyses, the heavy metals were analyzed as well as the organic constituents. A summary of these results is presented in Table 3. The full report from EAL Corporation, except for the GC scans, is presented in Appendix C.

The samples used in these analyses were taken from respective blends of wastes that were recovered from the cells that contained the membrane liner specimens at the time the cells were dismantled. The blends of the wastes from these cell were used because it was felt that they reflected the composition of the original wastes, though scavenging of some components might have taken place and some volatiles may have been lost.

It was recognized that the organics would be absorbed by the polymeric liner materials and that the degree of absorption would depend on the organic constituent and its concentration. The GC/MS data from EAL Corporation was reviewed for three of the wastes that contained organics, i.e. the lead waste, the pesticide waste, and the "Oil Pond 104" waste. The concentrations of some of the constituents that appeared on the GC/MS scan are presented in Table 3. Only two compounds, butyl benzene phthalate and dibutyl phthalate, were found in all three wastes. The oily hydrocarbon constituents, however, were not included which, in the case of the lead waste and the "Oil Pond 104" waste, are the major organic constituents. It would have been desirable to analyze the extractables in the exposed liners for these constituents but that was beyond the scope of the project.

HANDLING OF WASTES AND FILLING OF EXPOSURE CELLS

When the exposure cells were loaded with wastes, only five of the six types of wastes were available in sufficient quantities, i.e. 200 gal or four drums, to fill the desired number of cells:

- Acidic waste ("HNO_3-HF-HOAc"), W-9.
- Alkaline waste ("Spent caustic"), W-2.

TABLE 3. SUMMARY OF GENERAL PHYSICAL PROPERTIES AND HEAVY METALS CONTENT OF WASTES[a]

Phases and tests	Acidic "HNO_3-HF-HOAc" (W-9 Rec)	Alkaline "Spent caustic" (W-2 Rec)	"Lead waste blend[b]" (W-14 Rec)	Oily waste "Oil Pond 104" (W-5 Rec)	Pesticide "Weed Killer" (W-11 Rec)
Phase I, aqueous insoluble organic liquid, weight %	2.6	3.4	13.6	99.0	0.24
Phase II, aqueous, weight %	96.3	73.1	86.2	0.6	99.4
Phase III, solids, weight %	1.1	23.5	0.2	0.4	0.4
pH, aqueous phase	0.5	7.7	7.6	c	2.6
Specific gravity, aqueous phase	1.07	1.19	0.97	c	1.00
organic phase	c	c	0.79	0.92	c
Flash point, °C	None, boils	None, boils	<0°C	None, boils	None, boils
Metals analysis[d], mg/L:					
Antimony	9.6	50	87	500	9.0
Arsenic	0.19	<0.02	<0.02	<0.02	<0.02
Beryllium	<0.2	<0.2	<0.2	<0.4	<0.2
Cadmium	0.22	0.6	<0.76	8.0	<0.15
Chromium	22	<0.8	1.9	290	5.4
Copper	5.6	2.3	1.1	33	9.2
Lead	28	5.0	95	170	1.4
Mercury	<0.003	<0.003	0.06	1.6	<0.003
Nickel	9.0	1.8	0.9	4.0	24.5
Selenium	<1	<1	<1	<1	<1
Silver	<0.05	0.33	<0.07	12	<0.05
Thallium	0.21	0.03	<0.02	<0.02	<0.02
Zinc	42	8.2	64	450	150
Barium	5.0	19	4.0	<5	4.8
Cobalt	2.9	24	7.8	40	1.2
Molybdenum	0.8	2.0	0.44	0.1	0.5
Vanadium	<2.5	5.1	<2.7	10	<2.5
Titanium	19	12	8.5	230	3.0
Organolead	c	c	32	c	c
Boron	c	c	8.1	c	26

[a] Analyses performed by EAL Corporation, Richmond, CA, on blends of wastes recovered from exposure cells with polymeric membrane liners. See Appendix C for full report of EAL Corporation, except for GC/MS scans.

[b] Data combined for aqueous and organic phases.

[c] Not applicable or no data.

[d] Atomic absorption analysis.

- Lead waste blend, W-14.
- "Oil Pond 104" waste, W-5.
- Pesticide waste ("Weed killer"), W-11.

All of the wastes were received in closed-head, 55-gal drums and were much lower in viscosity than had been anticipated. It was originally expected that they would be sludge-like and could be handled with buckets from open-top drums. Furthermore, some were found to be very inhomogeneous, consisting of two liquid phases and particulate solids. Since the waste of a given type should have the same composition in all the cells in which it was placed, the waste from the different drums had to be thoroughly mixed and blended before it was added to the cells. Pumping the wastes from drum to drum and into the individual exposure cells appeared to be the best way to handle waste and reduce the chances for spillage. The equipment which was set up to mix and pump these wastes in shown in Figure 1.

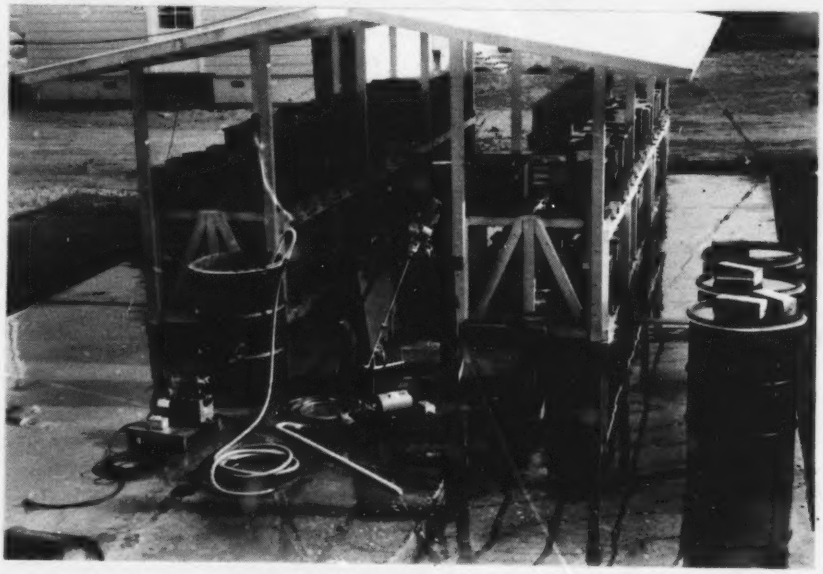


Figure 1. Equipment used in the blending of wastes and filling of cells.

It was necessary to obtain a pump which was compatible with the wastes, self-priming, able to pump solids in suspension, and had sufficient capacity to transfer the contents of a 55-gal drum in 10 minutes. A flexible impeller pump was obtained which had an epoxy pump body and a totally enclosed motor which allowed hosing down of the pump without getting water into the motor. The rated capacity was 7 gal per-minute. Both the impeller and the pump body were replaceable in the event either was damaged by the wastes. The plan for the mixing and blending of each waste was:

1. To mix thoroughly the contents of each drum by pumping them into an open-top drum equipped with a 1750 rpm mixer, with dual propellers and a polyethylene liner, and mixing with the remaining solids flushed from the original 55-gallon drum.

2. To transfer one-fourth of the contents to each of four additional open-top drums for blending.

3. To accumulate the mixed quantities in the open-top blending drums and mix each drum thoroughly.

4. To pump the wastes from the open-top blending drums into the individual cells.

Thus, each exposure cell received an aliquot of the waste of the same composition as that received by every other cell containing that waste. Five-gallon samples of the individual wastes were withdrawn for the analysis described above.

The wastes were loaded in the cells in the order of anticipated increasing difficulty, based on viscosity and the apparent amount of solids. The "Pesticide" having the consistency of water was processed first, without problems, using the flexible impeller pump.

The "Spent caustic" waste appeared to be the next easiest to handle, as it was less corrosive than the strong acid ("HNO_3-HF-HOAc") waste and appeared to be an aqueous solution. However, before two drums of the caustic had been mixed and transferred, undissolved salts wore away the epoxy body and faceplate of the pump and it ceased to operate. To solve this problem, a second pump, a centrifugal type of 20 gal per minute, was obtained. The impeller and housing are made of cast iron which was more resistant to the abrasion of the salts in the caustic waste than was the epoxy body of the first pump. Furthermore, the centrifugal pumping action did not depend upon close fitting parts and handled particles up to 3/8 in. in diameter.

A strainer was placed over the end of the inlet pipe to strain large particles when transferring from the closed-head drums. These large particles were eventually broken-up manually to a pumpable size, and flushed out of the drum. The shearing action of the mixer in the open-head drum further disintegrated these insoluble salts.

To fill the 5-gal individual exposure cells using the 20 gpm centrifugal pump, it was necessary to reduce the flow by putting a "T" in the system and circulating a portion of the waste. This pumping system worked very well with the caustic waste.

There was an insufficient amount of a single lead waste to fill the primary exposure cells; consequently, the three different lead wastes which were available were combined by the mixing and blending process described above to yield the four drums needed to fill the exposure cells. The centrifugal pump was used in the mixing, blending, and transferring of the Lead wastes.

The saturated and unsaturated oil waste was viscous and had to be pumped with the flexible impeller pump. Pumping and blending this oily waste was speeded slightly by warming it with a mantle drum heater.

As the flexible impeller pump is resistant to acids, it was used to pump the "HNO_3-HF-HOAc" waste. The acid would have attacked the cast iron of the centrifugal pump, changing both the character of the waste and of the pump.

The sixth primary waste, the slurry oil, was highly viscous and only two drums were available. The blending of the two was dropped and the cells were loaded directly after heating with electric blankets and homogenizing the contents of each drum separately.

All the remaining wastes or media were homogeneous and low in viscosity and posed no problem in handling.

SECTION 5

SELECTION AND PROPERTIES OF LINER MATERIALS

To meet the basic objective of this study of the effect on liner materials of contact with different hazardous wastes, a broad spectrum of potentially satisfactory materials was considered. These materials included compacted native clay soils, admixed materials, such as soil cements, asphaltic concrete and bentonite, sprayed-on membranes, and polymeric membranes, all of which had found wide use for lining canals, reservoirs, ponds, and other water-retention and conveyance facilities. Though the performance of all these materials with respect to water was well known, their performance and durability with respect to waste liquids containing dissolved inorganics, organics, and oils were uncertain.

The materials selected as liners for the primary exposure test were:

- One fine-grain native soil.
- Three admixed materials:
  - Asphalt concrete (hydraulic)
  - Bentonite/sand mixture
  - Soil-cement.
- One sprayed-on asphalt membrane.
- Eight commercial or developmental polymeric membranes.

The specific materials and the test methods used in characterizing each material are described in the following subsections. Also described is the preparation of the test specimens mounted in the cells.

COMPACTED NATIVE CLAY

A soil liner is made from soil material that is either on or near the waste disposal site. The soil is treated, remolded, and/or compacted so as to form a layer of low permeability. Because of their general availability and economic considerations, soils generally have been, and remain, the first material considered for lining a waste containment facility. The initial study of a site will usually include an assessment, based on engineering, environmental, and economic criteria, of the characteristics of the available native soil to determine whether or not it can be used to produce an effective liner for the particular waste to be contained.

Low permeability is the most important requirement of a soil to be used as a liner for a waste impoundment. However, in addition to low permeability, the soil liner also should adsorb the potentially polluting species, at least temporarily. Both low permeability and high adsorption capacity of a soil are often associated with the presence of soil fines. Thus, the proportion of clay size particles (less than 2 μm) is an important consideration when assessing a soil as a potential lining material. A specific limiting value of clay-size particles cannot be stated categorically because both soil permeability and adsorption capacity are dependent on other factors not directly related to the proportion of fines. These other factors include the gradation and the degree of weathering of the nonclay fraction and the physicochemical and mineralogical properties of the clay fraction. Depending on these properties and on the required permeability to water, which in general will be in the 10^{-9} to 10^{-6} cm s^{-1} range, the acceptable proportion of fines in a soil liner will most likely be greater than 15-20% of the soil mass.

Selection of Soil

Since only one soil was to be tested in this project, we felt that it should have as low a permeability as possible and should have minimal physicochemical interaction with the wastes. Several fine-grain soils were obtained and tested; they included:

1. A soil material used for "mudjacking" of portland cement concrete pavement.

2. A clay used for constructing tennis courts and baseball diamonds.

3. A soil used to line a waste pond at a power plant in Moapa, Nevada.

4. A soil material of fines which was a by-product of washing crushed graywacke sandstone and which had been used to construct a cover of low permeability over a chemical waste dump.

5. A very fine silt that had been dredged from the Carquinez Strait and the lower Napa River.

The last soil was reported to us as having low permeability and was obtained at the Mare Island Naval Shipyard, Vallejo, California.

These fine-grain soils were evaluated at different water contents in the laboratory by measuring the hydraulic conductivity of remolded specimens with a triaxial, back pressure permeameter (Vallerga and Hicks, 1968) to determine the minimum permeability that could be obtained with each soil. Soil samples were oven-dried and then allowed to come to equilibrium at room temperature, which took approximately 3 weeks. Test specimens were made by mixing 200 g of dried soil with predetermined amounts of tap water (Table 4) and compacted in a 2-in. diameter, 2-in. deep mold, using an impact-action hand drill with a 0.5-in. diameter foot, followed by 2

TABLE 4. CHARACTERISTICS OF WATER USED IN PREPARING SOIL AND ADMIXED LINERS

Constituent or property	Concentration, mg/L except pH and conductivity
Cations	
Calcium, Ca	6.0
Magnesium, Mg	0.6
Sodium, Na	1.3
Potassium, K	0.5
Anions	
Chloride, Cl	2.0
Carbonate, CO_3	1.1
Sulfate, SO_4	0.9
Nitrate, NO_3	0.05
Bicarbonate, HCO_3	18.0
Phosphate, PO_4	0.013
Other	
Hardness, total as $CaCO_3$	17.5
Alkalinity, as $CaCO_3$	17
Total dissolved solids	33
pH	9.1
Electrical conductivity, μmhos/cm	45

Source: East Bay Municipal Utility District, Oakland, California, December 1975.

minutes double-end static compaction at 7,000 lb. The permeabilities were determined with a back pressure of 1.0 atm, a confining pressure of 1.2-1.4 atm, and a gradient of around 25. The amount of time specimens were pressurized before a run varied. The results are shown in Table 5.

The fine silt dredged from Carquinez Strait, subsequently called Mare Island soil, was selected for exposure testing in this project. The permeability of undisturbed cores cut from the water-laid deposits was between 10^{-8} and 10^{-7} cm s^{-1} with a similar range found on laboratory-compacted specimens prepared at water contents ranging from 14% to 30% (Table 5).

Figure 2 shows that a small variation in permeability takes place over a considerable range of moisture contents. This was the first clue to the fact that, although clayey in terms of the size of the dominant particle fraction, the Mare Island soil is actually of very low concentration in active clay minerals. The minimal permeability was obtained around a moisture content of 22% which, assuming a 95% saturation and a specific density of 2.65 g cm^{-3}, indicates a soil bulk density of 1.64 g cm^{-3}.

Sieve analysis of the soil (ASTM D1140-54) showed more than 98% passed the 200 mesh sieve (75 μm) and almost 95% passed the 325 mesh sieve

TABLE 5. WATER PERMEABILITY OF LABORATORY COMPACTED NATIVE CLAY SOILS

Soil[a]	Water[b], parts per 100 dry soil	Coefficient of permeability[c]	
		cm s^{-1}	in. yr^{-1}
Tennis court clay	10	4.0×10^{-5}	497
Mudjacking soil	10	1.8×10^{-5}	223
Mudjacking soil	11	7.9×10^{-6}	98
Moapa, Nevada, #2	15	1.7×10^{-7}	2.10
Moapa, Nevada, #2	17	4.1×10^{-8}	0.51
Graywacke fines	12	4.9×10^{-6}	61.0
Graywacke fines	15	2.8×10^{-7}	3.5
Fine silt dredged from Carquinez Strait - "Mare Island Soil"	30	8.2×10^{-8}	1.02
	28	7.8×10^{-8}	0.97
	25	9.0×10^{-8}	1.12
	20	6.5×10^{-8}	0.81
	18	7.3×10^{-8}	0.91
	16	1.1×10^{-7}	1.37
	14	1.2×10^{-7}	1.49

[a] Carbonate content of soils: Graywacke fines soil contains some carbonate; Moapa soil contains more carbonate; Carquinez Strait silt contains no carbonate.

[b] Soils mixed with various amounts of water, compacted, and tested to determine water content to achieve minimum permeability.

[c] Measured with triaxial, back pressure permeameter (Vallerga and Hicks, 1968).

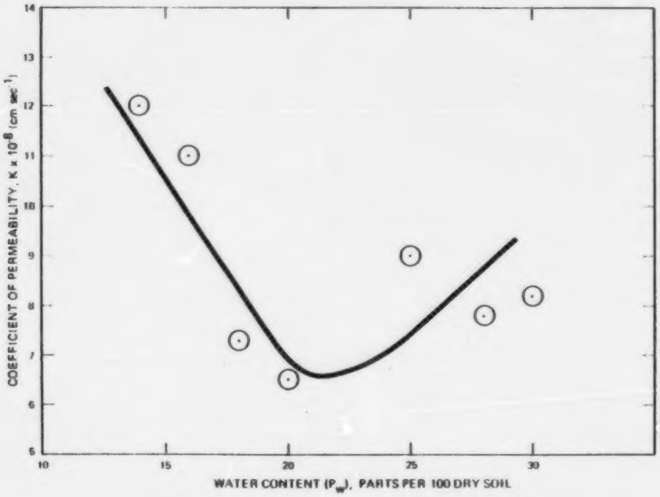


Figure 2. Relationship between moisture content (P_W) at compaction and saturated hydraulic conductivity (K) of Mare Island soil.

(45 µm). These results were consistent with particle-size data determined by hydrometer analysis (ASTM D422-63) which indicated that more than 50% of the soil mass was made up of clay-size particles (2 µm).

The consistency of the Mare Island soil was also determined. The liquid limit, using the standard procedure with Casagrande's apparatus (ASTM D42366), was found to have an average value of 65%. The lower plastic limit which averaged 47% was much more difficult to obtain because of the number of tests required for establishing an accurate value. The plasticity index (the difference between the plastic limit and the liquid limit) of the soil is 18%; thus, qualitatively the soil is slightly plastic. The activity of the clay fraction (the plasticity index divided by the percent of soil mass made up of clay-size particles) was very low, i.e. 0.36, which almost excludes the presence of any smectite type of minerals. This finding deserves a special discussion: when soils are chosen for waste-disposal sites, it is often considered an advantage to have a soil with a large proportion of smectite clay. Such a soil is considered good because the characteristic particle size of a smectitic clay is the smallest among clay mineral categories and thus the consensus is that a smectite clay should result in the liner of lowest permeability; the high cation exchange capacity of this clay should also help in temporarily retaining cationic species. While this may be the case, it is also true that an inert, nonsmectitic clay has the advantage of being resistant to physicochemical alterations which may lead to hydromechanical changes. In the case of the Mare Island soil, its low activity indicated the presence of nonactive clay; thus, the likelihood that the range of permeability found when water was the percolating solution would be maintained upon subsequent use of hazardous wastes. In the next subsection, we present experimental results that will prove this was the case.

The Mare Island soil had a high concentration of soluble salts (5.5 g/ 100 g dry soil) and a pH of 6.5.

Preparation of Compacted Soil Specimens

The soil for the exposure test specimens was compacted into 13-in.-high spacers, which were coated on the inside with primer, Epoxy 2, and sandblasting sand. The compacted soil measured 12 x 17 x 12 inches.

An attempt was made initially to compact the soil in a spacer which had been bolted onto the base, but the assembly was too flexible; the bottom of the base and the sides of the spacer flexed with each impact of a tamper. The bond between the soil and the spacer was not tight and it was felt that the lowest potential water permeability was not achieved. It was, therefore, decided to compact the soil in the spacer first and mount the specimens onto the base. A wooden frame was constructed (Figure 3) in which the spacer was held tightly to prevent bulging of the sides and the whole assembly was bolted to a concrete floor. A piece of polyethylene film was placed on the floor in the assembly before the soil was compacted. This film was removed before the liner was mounted in the base.

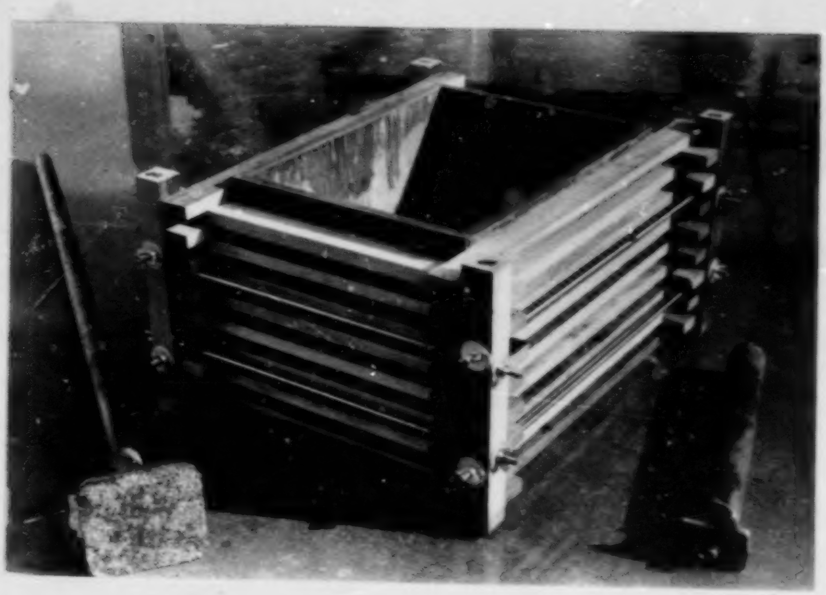


Figure 3. Jig used in compacting the specimens of soil and the other admix materials. It was bolted to the floor during compaction. The spacer for the soil specimens fit into this jig as assembled.

The three tampers were used in compacting this soil as well as the soil-cement and bentonite clay specimens:

Tamper 1: Weight 6.8 kg (15 lb)
Face, flat square, 125 x 125 mm (5 x 5 in.)

Tamper 2: Weight 7.3 kg (16 lb)
Face A, flat round, 50 mm diam. (2 in.)
Face B, "sheep's foot", 13 mm diam. (0.5 in.)

Tamper 3: Face, flat rectangular, 20 x 40 mm (0.75 x 1.5 in.)
Struck with 1 kg hammer (2 lb)

Because of its plastic nature, the Mare Island soil could not be blended with the soil-shredder. Near or above its optimum moisture content, the soil was too sticky to pass through the shredder, and, when drier, it was too hard causing the shredder to stall. Therefore, soil for the liners was blended by taking small scoopfuls from each bag of soil and discarding lumps that were dry or unusually wet. Soil for each lift (1.5 to 2 in. loose, compacted to 1 to 1.25 in.) was placed in the spacer, crushed, and leveled with Tamper 1 and compacted by repeated tamping with Tamper 2 using alternatively the 2-in. diameter flat surface "A" and the 0.5-in. diameter "sheep's foot" face B. Final compaction of each lift was with the "sheep's foot" face to provide a rough surface for keying to the next layer. The edges were compacted with Tamper 3 struck with a hammer twice around all edges for each lift. The top layer of each liner was leveled, after compacting, by additional tamping with Tamper 1. The compacted soil liner is shown in Figure 4. These liners were covered with polyethylene film and protected from drying until mounted in the cells.

The cell bases were filled with the silica gravel leveled to support the soil. After the liners were mounted in the cells, a ring of Epoxy 1 was cast into a triangular groove cut into the soil around the periphery of the liner. The outside of the joint between the spacer and the cell was sealed with butyl caulk.

Cores cut from compacted soil liners were slightly more permeable than the cores from the water-laid deposits removed from Mare Island. Results from measuring the permeability of cores from compacted liners are presented in Table 6.

ADMIXED LINER MATERIALS

The admixed liner materials were selected or designed to yield permeability coefficients of 10^{-7} cm s^{-1} or less. The admix materials selected for the exposure test are:

Hydraulic asphalt concrete	2.5 in. thick
Modified bentonite and sand	5.0 in. thick
Soil-cement with surface seal	4.0 in. thick

Though water permeability was the primary test in evaluating admix materials, other tests appropriate to the specific materials were also used. The tests used in evaluating the admix materials are discussed under each individual material.

Sources of materials for the admix liners are shown in Appendix E. Details regarding the individual liner materials are presented below.

Asphalt Concrete (Hydraulic)

The mix design used for the hydraulic asphalt concrete (HAC) liner specimens called for dense-graded aggregate to 0.25 in. maximum size and 9 parts of asphalt AR-4000 per 100 aggregate. A one-half-ton batch of hydraulic concrete was prepared in a hot-mix plant and hauled 5 miles in an insulated trailer to the test site. The temperature during mixing was 345°F. The hot concrete was raked into a form (10 ft x 3.5 ft x 2.25 in.) and leveled with a wooden screed. Compaction was started when the mix had cooled to 275°F and was performed with a gasoline-powered "Jumping Jack" rammer having a 10 x 10 in. compacting foot. Compaction was continued for one-half hour, at which time the HAC began to stick to the compactor foot. The HAC was quite plastic, and some asphalt bled to the surface.

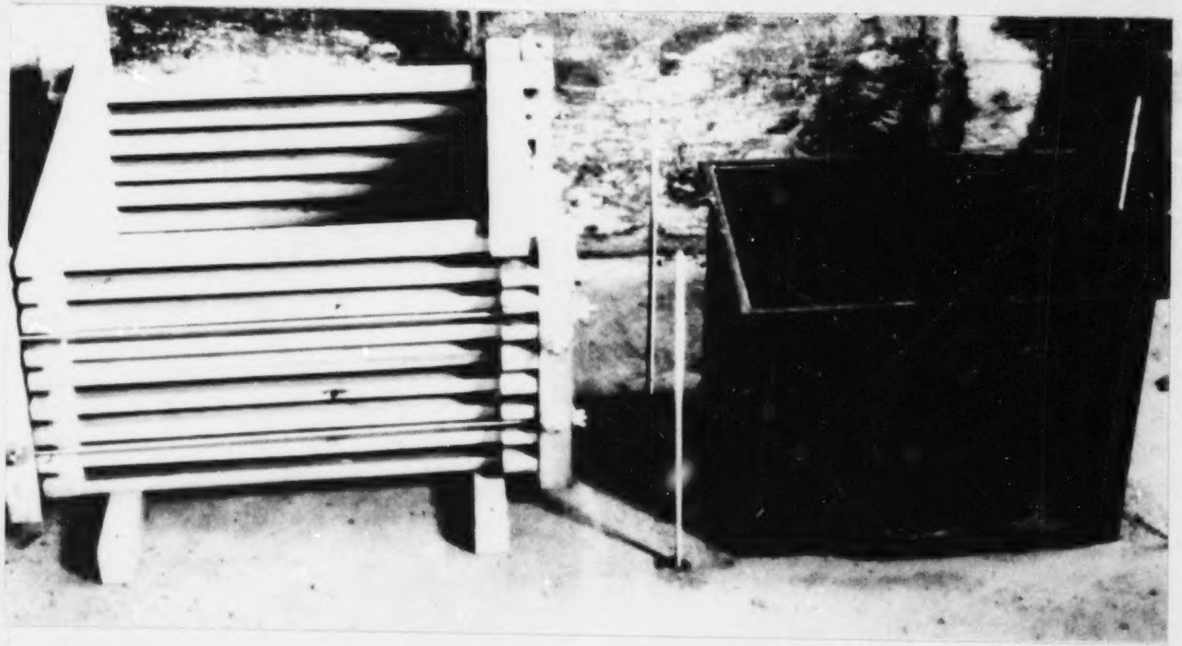


Figure 4. A compacted fine-grain soil liner specimen in 13-in. tall spacer ready to be mounted in cell base. Also shown are the frame in which the specimen had been compacted and bolts for holding the assembly to the floor while the liner was compacted.

TABLE 6. PERMEABILITY OF COMPACTED SOIL LINER SPECIMENS[a]

Description of specimen	Coefficient of permeability, cm s^{-1}
Laboratory-prepared specimens (7 samples)[b]	6.5×10^{-8} to 1.2×10^{-7}
Core from specimen compacted in spacer on cell base[c]	
Core from "resilient" end of cell	4.5×10^{-7}
Core from "firm" end of cell	1.7×10^{-7}
Core from 12-in. thick specimen compacted in spacer in jig on concrete floor (2 samples)	9.4×10^{-8} and 4.2×10^{-7}
Core taken from water-laid deposits of the soil (3 samples)	1.0×10^{-8} to 1.0×10^{-7}

[a] Permeabilities determined in a back pressure triaxial permeameter (Vallerga and Hicks, 1968) with a back pressure of 1.0 atm, a confining pressure of 1.3 atm, and a gradient of about 25.

[b] See Table 5.

[c] Due to the geometry of the cell base, the shallow end of the base was "springy," and the soil at that end rebounded during compaction.

A one-gallon sample of uncompacted HAC and a one-quart sample of the AR-4000 asphalt (from the tank at the mixing plant) were retained for laboratory tests. After the compacted HAC had cooled overnight, 14 two-inch diameter cores were cut.

The tests performed on the asphalt and the concrete are listed in Table 7 and the results are presented in Tables 8 and 9.

The water permeability of six of the cores ranged from 2.8×10^{-8} to 1.7×10^{-9} cm s^{-1} (Table 8). The compacted slab was cut with a diamond saw into 11.75 x 17 in. specimens to fit in the spacers for mounting in the cells. The consistencies of the original asphalt and extracted asphalts, as measured by penetration values, are presented in Table 9. These data show the hardening of the asphalt which takes place during mixing and compaction.

Each specimen was sealed into a 3.5-in. spacer. The specimen was first placed on a cell base which had been filled with silica gravel. A spacer was placed around the specimen and a wax-coated metal form was placed on top of it to form a ring. The space between the spacer, the liner, and the base was filled with a grout consisting of Epoxy 1 (2 Part A:1 Part B:3 sand). Grout of a similar composition was used to cast the ring above the specimen.

TABLE 7. TESTING OF HYDRAULIC ASPHALT CONCRETE

Property	Test method	Original	Exposed
Water permeability	Back-pressure permeameter (Vallerga and Hicks, 1968)	x	x
Density and voids	ASTM D1184 and D2041	x	x
Water swell	California Division of Highways 305	x	...
Compressive strength	ASTM D1074	x	x
Asphalt content	ASTM D1856	x	x
Penetration of asphalt	ASTM D5	x	x
Viscosity of asphalt, sliding plate	California Division of Highways 348	x	x
Sieve analysis of the aggregate	ASTM C136 and C117	x	...

TABLE 8. WATER PERMEABILITY OF CORES CUT FROM SLAB OF HYDRAULIC ASPHALT CONCRETE COMPACTED FOR PREPARING LINER SPECIMENS[a]

Core	Location on slab	Coefficient of permeability	
		cm s^{-1}	in. yr^{-1}
5	In area compacted last	6.6×10^{-9}	0.08
7	In area compacted last	2.8×10^{-8}	0.34
12	In area compacted last	2.1×10^{-8}	0.26
16	In area compacted first	$<1 \times 10^{-8}$[b]	<0.12[b]
	Top slice removed	1.7×10^{-8}	0.21
17	In area compacted first	$<1.5 \times 10^{-8}$[b]	<0.19[b]
18	In area compacted first	ca 1.7×10^{-9}	ca 0.02

[a] Permeabilities measured in a triaxial, back pressure permeameter with a pressure of 1.0 atm, a confining pressure of 2.0 atm, and a gradient of about 25.

[b] Because of long test runs, the specimens were removed from test before constant values were obtained.

TABLE 9. PROPERTIES AND COMPOSITION OF HYDRAULIC ASPHALT CONCRETE

Property	Unexposed concrete/asphalt 1975/76	Unexposed concrete/asphalt 1983
Properties of concrete		
Density, $\mathrm{g\ cm^{-3}}$	2.322	2.391
$\mathrm{lb\ ft^{-3}}$	145.0	149.3
Voids content, % by volume	4.0	1.3
Voids ratio (vol voids/vol solids), %	4.2	1.4
Compressive strength		
Initial, psi	486[a] (268[b])	...
After 24 h immersion in water, psi	360[a]	...
Retention of strength after immersion, %	74[a]	...
Coefficient of permeability, $\mathrm{cm\ s^{-1}}$	1.3×10^{-8}	...
Analysis of concrete[c]		
Asphalt extracted, %	7.8	7.7
per 100 aggregate	8.5	8.3
Sieve analysis of aggregate, % passing:		
1/2 in. (12.5 mm)	100	...
3/8 in. (9.5 mm)	98	...
1/4 in. (6.3 mm)	95	...
No. 4 (4.75 mm)	84	...
No. 8 (2.36 mm)	52	...
No. 16 (1.18 mm)	34	...
No. 30 (0.60 mm)	23	...
No. 50 (0.30 mm)	17	...
No. 100 (0.15 mm)	13	...
No. 200 (0.075 mm)	10	...
Soluble in hydrochloric acid[d], %		
Rock, >No. 8 (>2.36 mm)	0.54	...
Sand, No. 8 to No. 200 (0.075 to 2.36 mm)	1.16	...
Silt, <No. 200 (<0.075 mm)	4.59	...
Asphalt properties		
Penetration at 25°C, 100 g, 5 s		
Unmixed asphalt from tank at hot mix plant	138	...
Extracted asphalt at 25°C	80	78
Asphalt extracted from uncompacted HAC	96	1
Viscosity at 25°C (sliding plate viscometer), poise		
at $0.05\ \mathrm{s^{-1}}$		1.47×10^{6}
at $0.01\ \mathrm{s^{-1}}$		1.78×10^{6}
at $0.001\ \mathrm{s^{-1}}$		2.36×10^{6}
Shear susceptibility at 25°C		0.12
Calculated penetration at 25°C		74

[a] Determined in December 1976 on cylinders molded November 1976 from uncompacted HAC retained in closed cans since August 1975; average of three tested dry, three tested after immersion.

[b] Determined in November 1976 on one core cut from HAC compacted in August 1975.

[c] Design: Dense-graded aggregate (0.25-in. max) 100; asphalt (AR4000, ASTM D3381) 9.0.

[d] AWWA Standard B-100-72-72 Section 5.3.

The HAC liner specimens in the spacers were mounted in the cells and were tested with water prior to adding wastes (Figure 5). Five were found to leak between the spacer and the epoxy seal, probably the result of differences in thermal coefficients of the steel and the asphalt concrete. Five of the asphalt concrete specimen-spacer combinations were chilled and the steel spacers were removed. The surfaces of the original epoxy seal were roughened and the new spacers of epoxy resin-aggregate mix were cast around and on top of the epoxy seal. This was done through the use of a wax-coated steel form and a wooden outer form. The cast epoxy spacers consisted of Epoxy 2 filled with sand (2 Part A:1 Part B:4.5 sandblasting sand) with a thin-layer of unfilled Epoxy 3 poured on top to form a level surface. Neoprene sponge gaskets were used to seal the joints between the spacers and exposure cell tanks as in all the other cells.

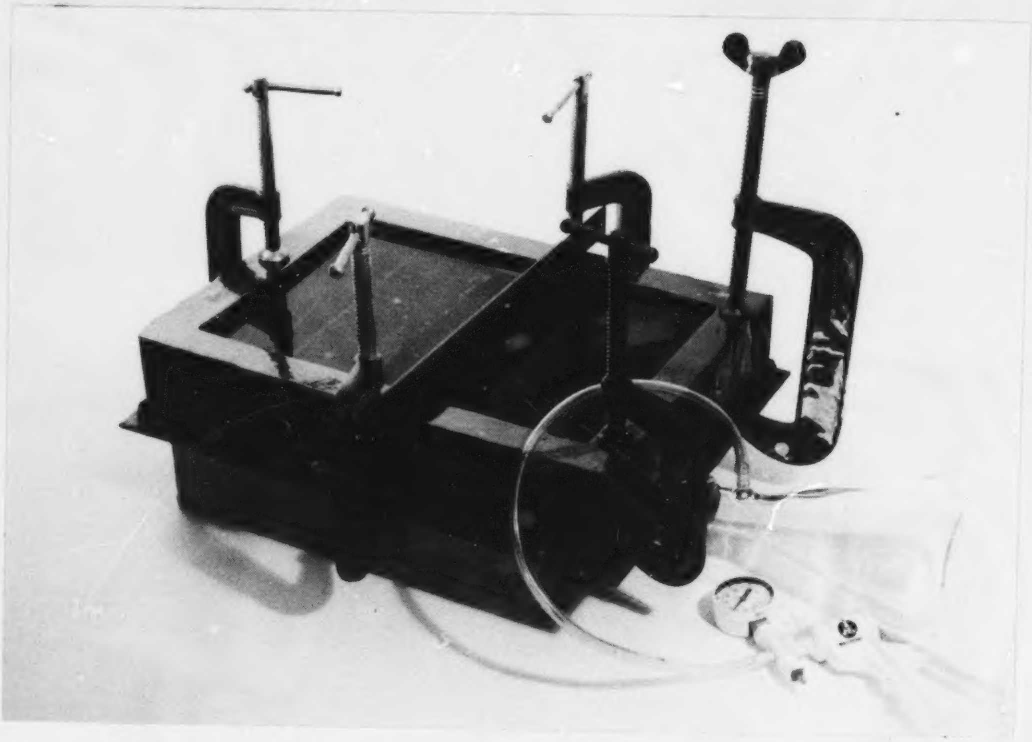


Figure 5. Leak-testing of hydraulic asphalt concrete. Specimen is covered with water and the base pressurized.

Bentonite-Sand Admixes

Bentonite is a colloidal clay composed chiefly of the clay mineral smectite (montmorillonite). Two major varieties of bentonite that have commercial application are: (1) sodium bentonite, which has a high swelling capacity in water, and (2) calcium bentonite which has a negligible swelling capacity. Because of its high swelling capacity, sodium bentonite is used as a sealant or a lining material for water storage and conveyance. Polymer-modified bentonites have been developed, the suppliers of which have claimed improved resistance to salt contamination and to other contaminants aggressive to bentonites.

Bentonite is commercially available in bags or in bulk as a fine powder or as granules. When used as a lining material, it is either applied directly, or mixed into sand or the top layer of soil and compacted. In either case, the layer containing bentonite is generally covered with a protective soil cover. Slurry trenches, filled with soil-bentonite slurry, are used to control lateral movement of water or liquid wastes. Compacted soil-bentonite or sand-bentonite liners are often 4 to 6-inches thick.

Selection--

Two polymer-modified sodium bentonites were included in the study. The materials are hereafter referred to as Bentonite A and Bentonite B. Laboratory specimens were prepared with 6%, 12%, and 20% of each treated bentonite in a well-graded sand, and briquets were compacted with various water contents to determine optimum moisture content for compaction to maximum density. Sieve analysis of the sand used was:

Sieve size		
Mesh	Opening, μm	% Passing
8	2360	96
16	1180	80
30	600	50
50	300	15
200	75	2.6

As the sand-bentonite compositions were not strong enough to be tested without support, specimens at the optimum moisture content were compacted in brass tubes for testing in the back-pressure permeameter. Permeability coefficients were in the range of 10^{-7} to 10^{-10} cm s^{-1}, as shown in Table 10.

TABLE 10. WATER PERMEABILITY OF LABORATORY COMPACTED MODIFIED BENTONITE-SAND SPECIMENS

Composition			Coefficient of permeability, cm s^{-1}	
Sand	Water	Modified bentonite	Bentonite A	Bentonite B
94	12	6	5.0×10^{-7}	7.1×10^{-8}
88	12	12	7.4×10^{-9}	2.8×10^{-9}
80	12	20	8.0×10^{-10}	3.5×10^{-9}

Preparation of Polymer-Modified Bentonite-Sand Liner Specimens--

The liner specimens of polymer-modified bentonite were compacted in the 7-in. high spacers. Each spacer was placed on a cell base previously coated with Epoxy 2; the outer flange of the base was slightly roughened with sandpaper to ensure good adhesion of the epoxy seal to the epoxy coating. Masking tape was pressed around the flange between spacer and base, and a wax-coated metal form was placed on the base inside the spacer. Between the form and the spacer, an epoxy seal (approximately 0.5 in. high by 1 in. wide) was cast of grout made with Epoxy 2 and sandblasting sand. After the seal was cured and the form removed, the base was filled with crushed silica gravel to the level of the bottom of the seal. The gravel was covered with woven glass fabric and the wooden reinforcing frame was bolted around the spacer. The treated bentonite-sand mixtures were compacted in the spacer following the same procedure and with the same tampers used in compacting the fine-grain soil.

Eight liner specimens were fabricated of 12% Bentonite B and 88% well-graded sand, mixed at optimum moisture content and compacted in the liners already sealed to bases. For each liner, 53.5 lb of sand containing 3% water was mixed in a mortar boat with 5.5 pounds more water and with 7.1 lb Bentonite B. This mixture was placed in the prepared spacer in 1-in. lifts, and each lift was compacted with tampers. The compacting effort was less than that used for the native soil and soil cement liners in order to avoid damage to the epoxy seal between the spacer and the base. Two liner specimens were fabricated in the spacers of 20% Bentonite A and 80% well-graded sand mixed at 3% water content. The compacted Bentonite B liners were 5-in. thick and the dry-tamped Bentonite A liners 4-in. thick. Both were expected to be about 6-in. thick after complete hydration.

Two gallons of tap water was added on each liner through a perforated can to avoid displacing the tamped sand-clay mixtures. After one day, thickness increased from 4.0 to 4.5 in. and after one week to 4.75 in. One week was allowed for "fresh-water hydration" in accordance with the manufacturers' recommendations before draining the excess water and adding wastes. A sealing ring of Epoxy 2 grout was cast around the top edge of each of the compacted specimens. The average actual thickness after complete hydration was 5.2 in. for the Bentonite A liners and 5.5 in. for the Bentonite B liners.

Soil-Cement

Selection of Soil--

Most of the fine-grain soils being considered for use as compacted native soil liners were evaluated in soil-cement mixes. Formulations containing various proportions of soil:portland cement:water were molded into briquets, cured, and tested in the back-pressure permeameter. The lowest permeability was obtained with soil-cement based on "waste fines" from a local quarry, with 12 parts of Type 5 (sulfate-resistant) portland cement per 100 parts of soil, and 12% of water on the total of soil plus cement (Table 11). The "waste fines" material is available in large

TABLE 11. WATER PERMEABILITY OF SOIL-CEMENT SPECIMENS[a]

Soil	Type V cement, parts per 100 g dry soil	Water, parts per 100 g dry soil	Coefficient of permeability cm s^{-1}	Coefficient of permeability in. yr^{-1}
Tennis court clay	8	10	1.6×10^{-6}	20
Tennis court clay	10	10	1.3×10^{-6}	16
Tennis court clay	12	10	5.1×10^{-6}	63
"Mudjacking" clay	8	12	3.4×10^{-6}	42
"Mudjacking" clay	10	12	5.3×10^{-6}	66
"Mudjacking" clay	12	12	6.5×10^{-6}	81
Graywacke fines soil	10	13	1.9×10^{-6}	24
Graywacke fines soil	12	12	1.5×10^{-7}[b]	1.9[b]
Graywacke fines soil	10[c]	12	2.9×10^{-7}[d] 4.0×10^{-7}[e]	3.6 5.0
Core from specimen compacted in spacer in cell base	12	13.4	5.7×10^{-8}	0.71

[a] Except where otherwise noted, permeabilities determined in a back pressure permeameter with a confining pressure of 2.0 atm for all specimens except those made with "mudjacking" clay (1.3 atm confining pressure), a back pressure of 1.0 atm, and a gradient of approximately 25.

[b] Average of measurements with back pressures ranging from 1.0 to 3.0 atm.

[c] Rice hull ash cement (an acid-resistant pozzolanic cement).

[d] Average of measurement of back pressures ranging from 2.0 to 4.0 atm.

[e] Repeat with back pressure of 1.0 atm.

quantities as a by-product of the washing of crushed graywacke and has been used as a compacted soil cover over a chemical waste dump. Sieve analysis (wash) of the soil was:

Sieve size Mesh	Sieve size Opening, μm	% Passing
50	300	98
100	150	93
200	75	83
325	45	70

The graywacke fines soil was received mostly in large friable lumps that were broken up by passing the soil twice through a shredder to yield a thorough blend with maximum agglomerate size of approximately 0.125 in. (3 mm). The soil was air-dried on a concrete slab then stored indoors until used.

Preparation of Compacted Soil-Cement Liners--

The soil-cement specimens were compacted in 5-in. high spacers, the inner surfaces of which were coated with epoxy and sand. The compaction procedure was the same as used with the fine-grain soil; the spacer was placed within a supporting wood frame (Figure 6) to prevent bulging of the sides and the frame and spacer were bolted on a concrete floor. A piece of polyethylene film was placed on the concrete floor at the bottom of the spacer.

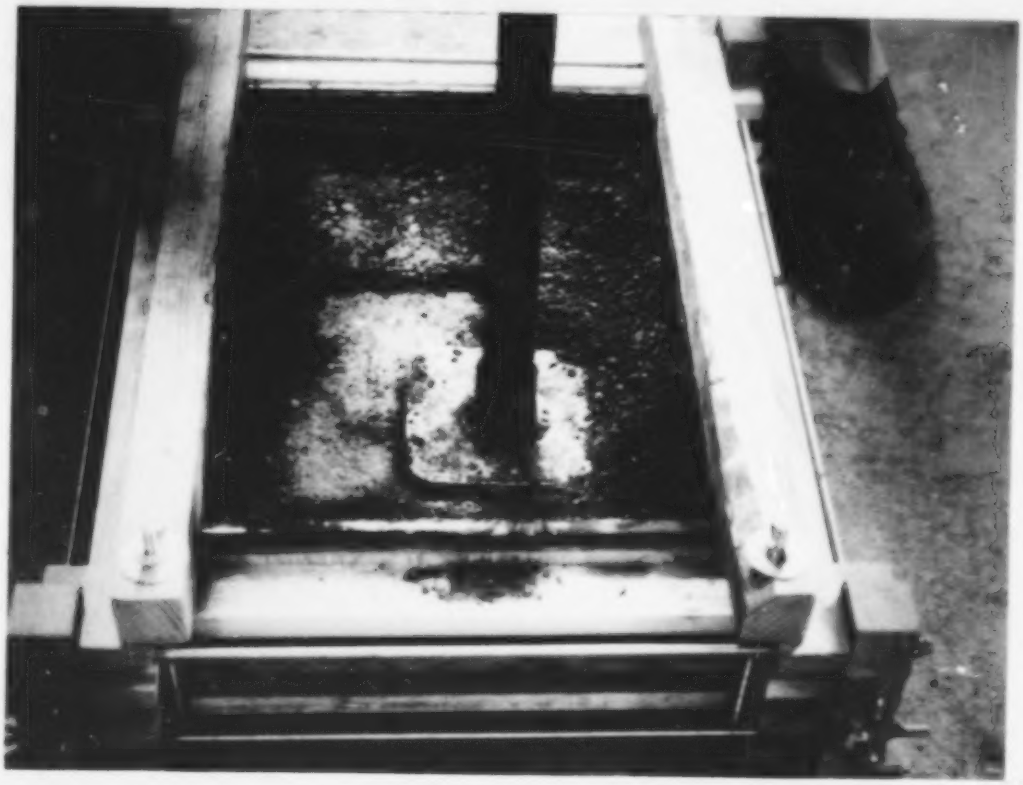


Figure 6. Compaction of a soil-cement liner specimen with Tamper 1 is shown as is the character of the uncompacted soil-cement.

For each liner, 50 lb of dry soil was thoroughly mixed in a mortar boat with 6 lb of Type 1-2-5 portland cement, then with 6.7 lb of tap water. The mixed soil-cement was crumbly and barely damp before compaction. One-inch lifts were placed in the spacer and compacted with Tampers 1, 2, and 3, described previously. Figure 6 shows the compaction of the soil-cement with Tamper 1. Final compaction of each lift was with the "sheep's foot" Face B of Tamper 2, to provide a rough surface for keying to the next lift. The edges were compacted with Tamper 3, twice around all edges for each lift. After a total compacted thickness of 4 in. was obtained, the top surface was leveled by additional tamping with Tamper 1. The compacted soil-cement was quite firm, but plastic enough to be compacted without cracking or shattering. The thickness of the compacted liner specimens was 3.75 to 4.0 inches. One-half gallon of water was poured on the surface and the soil-cement was cured for at least 7 days.

The surfaces of the cured liners were allowed to dry sufficiently to provide good adhesion for 1) an epoxy ring cast around the periphery of the liner, 2) sealing the liner to the spacer, and 3) application of two coal-tar modified epoxy surface sealing coatings to portions of the liner surfaces. After the epoxies cured, the liners were again flooded with water until they were filled with wastes. Each spacer was mounted on a cell base which had been filled with crushed silica gravel leveled to support the liner. Neoprene sponge gaskets were used to form seals between the flanges of the spacers and the cell tanks. The cell base, the spacer with sealed-in liner specimen, and the exposure tank were bolted together, and a bead of butyl caulk was applied to the outside of the base-to-spacer junction to make it airtight.

Application of Surface-Sealing Coatings--

Two sections of each soil-cement liner were coated with two coal-tar epoxy resin sealers. Seal-coats have been applied to soil-cement pond liners to increase strength, to increase resistance to wastes, and to reduce permeability. However, any settling and cracking in a soil-cement liner of a waste pond results in the cracking of the seal-coat. Preliminary tests indicated that both coatings under test were relatively brittle. A crack in the seal-coat would allow the waste to begin to attack the possibly weak interface of the seal-coat and the soil-cement. The exposed seal-coat edge on the soil-cement test liners would show if this interface was susceptible to attack. These two sealers and the primer used for one of them were two-component systems. Each was mixed according to the manufacturer's directions and applied by brush. The areas coated were 6-in. square, located at one end of the cell, as shown in Figure 7.

Coating K consisted of 3 coats of a proprietary epoxy bituminous coating. The first coat was diluted, 2 parts resin and 1 part thinner, according to manufacturer's instructions; the average total thickness was 0.044 in. Coating C consisted of 1 coat of primer, 0.007-in. thick, plus 2 coats of a second proprietary epoxy coal-tar coating, 0.018-in. thick, for a total thickness of 0.025 in.

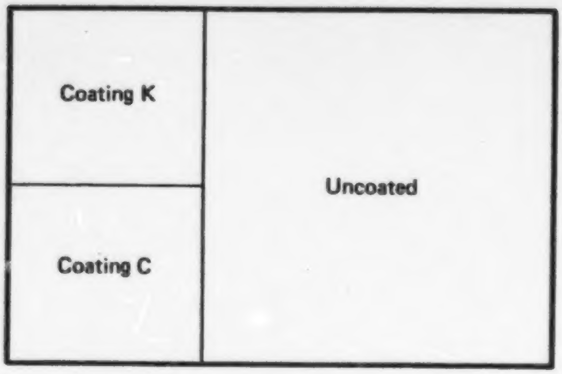


Figure 7. Pattern used in coating the soil-cement.

SPRAYED-ON ASPHALT MEMBRANE

Specimens of emulsified asphalt applied on a nonwoven polypropylene fabric mat were furnished by the supplier in 18 x 24 in. sheets, 0.3-in. thick, with a seam running across the width at the center. These sheets were cut to a size which would fit loosely inside the 1.25-in. spacer.

When mounting this liner, the cut sheet was placed on a base filled with crushed silica gravel; the spacer was placed around the liner, and an epoxy ring was cast around the inside of the spacer. The epoxy resin was spread on, around, and slightly under the edge of the liner. This ring of epoxy seals the liner to the base and the spacer, so that waste cannot by-pass the liner. The epoxy ring was a grout consisting of sand-filled epoxy resin (Epoxy 1:2 Part A:1 Part B:3 sand, and 2% thixotropic agent). The assembled cells were leak tested by filling with water; none of the seals leaked.

Properties of the asphalt sheet and of the extracted asphalt are presented in Table 12.

TABLE 12. PROPERTIES OF EMULSIFIED ASPHALT MEMBRANES

Property	Value
Moisture content of liner, %	0.26
Moisture vapor transmission, metric perm cm	6.7×10^{-2}
Viscosity[a] of extracted asphalt at 25°C, mP:	
At shear rate of $0.05\ s^{-1}$	6.1
At shear rate of $0.01\ s^{-1}$	5.9
At shear rate of $0.001\ s^{-1}$	5.7
Shear susceptibility	-0.02
Penetration at 25°C (calculated from viscosity)	41

[a] Sliding plate viscometer.

POLYMERIC MEMBRANE LINERS

When this project was initiated in 1975, flexible polymeric membranes were assuming increased importance as liner materials because of their very low permeability to water and other fluids. Polymeric membranes had been successfully used for the lining of water conservation and conveyance structures since about 1958; consequently, there was considerable interest in the use of such materials for the lining of waste storage and disposal facilities.

Polymeric membrane liner technology was then and remains (in 1983) relatively new, particularly with its application to waste containment. The current state of liner technology is described in the EPA Technical Resource Document, SW-870 (Haxo et al, 1983). The polymeric membrane liner materials vary considerably in polymer types, physical and chemical properties, methods of installation, costs, and interaction with various wastes. Not only are there differences among the polymers used, but also there are considerable variations in the lining materials of a given polymer type due to compound formulation, construction, and manufacturing differences among the producers.

Polymers used in the manufacture of lining materials include a wide range of rubbers and plastics differing in basic characteristics, e.g. chemical composition, polarity, chemical resistance, and crystallinity. They can be classified into four types:

- Rubbers (elastomers) which are vulcanized, i.e crosslinked (XL).
- Thermoplastic elastomers, which do not need to be vulcanized (TP).
- Thermoplastics which are generally unvulcanized (TP), such as PVC.
- Thermoplastics which have a relatively high crystalline (CX) content, such as the polyolefins.

Table 13 lists the various types of polymers that are used and indicates whether they are used as a thermoplastic, partially crystalline, nonvulcanized or vulcanized compound and whether fabric is used to reinforce lining materials manufactured with them. The polymeric materials most frequently used in liners at the time this project was undertaken were polyvinyl chloride (PVC), chlorosulfonated polyethylene (CSPE), chlorinated polyethylene (CPE), butyl rubber (IIR), ethylene propylene rubber (EPDM), and neoprene (CR). The thickness of polymeric membranes for liners has ranged from 20 to 80 mils, with most in the 20- to 60-mil range.

Note: Subsequently, liners based on high-density polyethylene (HDPE) were introduced and are being used in lining land storage and disposal facilities.

Most polymeric lining materials are based on single polymers and generally are compounded with fillers, antidegradants, plasticizers, and if crosslinking is needed, curatives. Compounds based on the same polymer can

TABLE 13. POLYMERS CURRENTLY (1983) USED IN MEMBRANE LINER MANUFACTURE

Polymer	Abbreviation	Type of compound[a]	Fabric reinforcement
Butyl rubber	IIR	XL	With and without fabric
Chlorinated polyethylene	CPE	TP and XL	Usually without fabric
Chlorosulfonated polyethylene	CSPE	TP and XL	Usually with fabric
Elasticized polyolefin	ELPO	TP (CX)	None used
Elasticized polyvinyl chloride	EPVC	TP	Usually with fabric
Epichlorohydrin rubber	ECO	XL	With and without fabric
Ethylene propylene rubber	EPDM	XL and TP	Usually without fabric
Neoprene (chloroprene rubber)	CR	XL	Usually without fabric
Nitrile rubber	NBR	TP and XL	With and without fabric
Polyethylene			
- High density	HDPE	CX	None used
- Low density	LDPE	CX	None used
Polyvinyl chloride	PVC	TP	Usually without fabric

[a] CX = crystalline, TP = thermoplastic, XL = crosslinked.

vary considerably in composition from manufacturer to manufacturer. Blends of two or more polymers, e.g. plastic-rubber alloys, are being developed and used in liners. Consequently, it is becoming difficult to make generic classifications based on individual polymers, although one polymer will generally predominate in the compound. Blending of polymers introduces the long-range possibility of the need for performance specifications. However, long-term liner performance in the field cannot, at the present time, be completely defined by current laboratory tests.

Most of the membrane liners currently manufactured are based on unvulcanized or uncrosslinked polymeric compounds and thus are thermoplastic. Even though a compound may be more chemically resistant once vulcanized, such as is the case with CPE and CSPE compounds, the lining material has been supplied unvulcanized because it is easier to seam and repair such a liner in the field. Noncrystalline, thermoplastic polymer compositions can be heat-sealed or seamed with solvent or bodied solvent (a solvent containing dissolved polymer to increase the viscosity and reduce the rate of evaporation). Semicrystalline sheetings, which are also thermoplastic, can only be seamed by thermal or fusion methods. Information on individual polymers and liners is presented in subsequent subsections. Subjects covered on each polymer are: composition, general properties and characteristics, general use, and specific use in liners.

In 1975, when making the selection of specific membrane materials for exposure tests, the following considerations served as guidelines:

1. To include a broad representation of polymer types because of the great diversity in the composition of wastes that may be impounded.

2. To include several polymeric membranes that were under investigation for resistance to MSW leachate (Haxo et al, 1982) which offered promise as liners for use in impounding hazardous wastes and allowed an intensive study of the characteristics of a few liners.

3. To exposure test liners of equal thickness. Most of the liners were available in 30-mil thickness; however, if this thickness was not available in the specific polymer type, then the thickness normally manufactured was used.

4. To test primarily the effect upon the barrier compound, that is, to test sheetings that were not reinforced with fabric.

In making the selection it was not possible to obtain liners from all liner producers, nor was it possible to obtain unreinforced samples of all polymer types. Representative liners of the different types were selected from those that were available. If several membranes of a given polymer were available, the membrane exhibiting the best physical properties was generally selected. Specific combinations of membranes and wastes were selected after preliminary screening tests which are described in Section 6.

Selection of Polymeric Membranes for Exposure Testing

The specific polymer types selected for the primary exposure in this study are listed in Table 14 and are discussed below in alphabetical order along with several other polymers that were tested in other exposures. Detailed data on the properties of these materials are given in Appendixes F and G.

Since 1975 there have been important developments in liner technology which led to the introduction of new polymeric compositions, compounds, and constructions into the market. The most important of these developments are included in the discussion below.

Butyl Rubber (IIR)--

Butyl rubber is a copolymer of isobutylene and a small amount of isoprene (2-5%) introduced in the polymer chain to furnish sites for vulcanization (crosslinking). Properties of butyl rubber vulcanizates that relate to their use as a liner are:

- Low gas and water vapor permeability.
- Thermal stability.
- Ozone and weathering resistance.
- Chemical and moisture resistance.
- Resistance to animal and vegetable oils and fats.

Butyl rubber is compounded with fillers and oil and generally vulcanized with sulfur. Some recent butyl compounds contain minor amounts of EPDM to improve ozone resistance. Butyl vulcanizates are highly swollen by hydrocarbon solvents and petroleum oils, but are only slightly affected by

TABLE 14. PRIMARY POLYMERIC MEMBRANE LINERS EXPOSED IN CELLS

Polymer	Liner number[b]	Thickness[a], mil	Type of compound[c]
Butyl rubber (IIR), fabric-reinforced	57R	34	XL
Chlorinated polyethylene (CPE)	77	32	TP
Chlorosulfonated polyethylene (CSPE), fabric-reinforced	6R	34	TP
Elasticized polyolefin (ELPO)	36	22	CX
Ethylene propylene rubber (EPDM)	26	50	XL
Neoprene (CR), fabric-reinforced	43R	32	XL
Polyester elastomer (PEEL)	75	8	TP
Polyvinyl chloride (PVC)	59	30	TP

[a] Nominal thickness.

[b] Matrecon serial number assigned to membrane liner material when received; R = fabric-reinforced.

[c] XL = Crosslinked or vulcanized; TP = thermoplastic; CX = partially crystalline.

oxygenated solvents and other polar liquids. Butyl vulcanizates also have a high resistance to mineral acids and a high tolerance for extremes in temperature, and retain their flexibility throughout their service lives.

Liners of butyl rubber were among the first synthetic liners to be used (Dedrick, 1980; Lauritzen, 1967; Smith, 1980). As of 1975, butyl rubber liners had been in service for about 25 years in water impoundments. In outdoor exposure in water management use, butyl rubber sheetings have generally shown little or no degradation after 20 years of service. These sheetings are generally seamed with two-part, low temperature curing adhesives which, primarily because of their lower degree of ultimate crosslinking, may be less resistant to the service conditions than the liner itself.

The particular butyl rubber sheeting selected for exposure in the primary exposure cells was vulcanized, had a thickness of 34 mil, and was reinforced with a nylon fabric of 22 x 11 ends per inch thread count. A second butyl sheeting of 62 mil, with no fabric reinforcement, was also selected for exposure in immersion tests.

Chlorinated Polyethylene (CPE)--

Chlorinated polyethylenes form a family of flexible thermoplastic polymers produced by chlorinating high-density polyethylene and, as such, they are saturated polymers with good aging and chemical resistance. In 1975, when this study was initiated, CPE liners had only recently been developed. CPE can be crosslinked with peroxides, but in membrane liners uncrosslinked thermoplastic compositions are generally used. CPE is compounded with fillers, such as carbon black and inorganic fillers, and with plasticizers. Membranes of CPE are seamed thermally with solvent adhesives or by solvent welding.

Note: In recent compounding practice, polyvinyl chloride or CSPE are sometimes added to improve tensile and thermal properties.

Most CPE compositions withstand ozone, weathering, and ultraviolet light, and resist many corrosive chemicals, hydrocarbons (if crosslinked), microbiological attack and burning. Also, some compounds of CPE are serviceable at low temperatures and are nonvolatile. As of 1975, sheetings of CPE had been used as linings for water reservoirs and for pits and ponds for storage of waste waters and chemicals.

The CPE membrane selected for the primary exposure and other exposure tests was a thermoplastic of 30 mil nominal thickness, and was not reinforced with fabric. Two other CPE sheetings were included in the study in immersion and other tests; one was a thermoplastic of 22-mil thickness and the other was a vulcanized sheeting of 36-mil thickness.

Chlorosulfonated Polyethylene (CSPE)--

Chlorosulfonated polyethylenes form a family of saturated (no double bonds in the polymer chain) polymers prepared by treating polyethylene in solution with a mixture of chlorine and sulfur dioxide. These polymers contain from 25-43% chlorine and from 1.0-1.4% sulfur and can be used in both thermoplastic (uncrosslinked) and in vulcanized (crosslinked) compositions.

Most CSPE compositions are characterized by ozone resistance, light stability, heat resistance, good weatherability, and resistance to deterioration by corrosive chemicals, e.g., acids and alkalies. They have good resistance to growth of mold, mildew, fungus, and bacteria, but only moderate resistance to oil. Membrane liners based on this type of polymer are available in both vulcanized and thermoplastic forms, but primarily in the latter. The thermoplastic versions slowly crosslink on exposure to moisture and the weather after placement. Usually CSPE sheetings are reinforced with a polyester or nylon scrim. They generally contain at least 45% of CSPE polymer by weight (Burke, 1979; Du Pont, 1973). The fabric reinforcement gives needed tear strength and dimensional stability to the sheeting for use on slopes; it reduces the distortion resulting from shrinkage when placed on the base and when exposed to the heat of the sun.

CSPE membranes can be seamed by heat sealing, dielectric heat sealing, solvent welding, or by using a "bodied" solvent adhesive. As of 1975, they had been used to line pits, ponds, and lagoons, and also to impound highly acid-contaminated fluids. After PVC membranes, CSPE membranes were the most widely used of the polymeric flexible liner materials.

The specific CSPE sheeting selected as the primary liner material for this study was a fabric reinforced nylon sheeting of 8 x 8 epi thread count and a thickness of 34 mils. It had been previously used as the primary CSPE liner in a study of liner materials in contact with MSW leachate (Haxo et al, 1982). Three additional CSPE sheetings were incorporated in the exposure test program. Two unreinforced sheetings were exposed in immersion and pouch tests and the third, a fabric-reinforced sheeting, was exposed as a primary liner specimen for two cells set up for immersion tests.

Note: None of the sheetings in the program was industrial grade; sheetings of this type were commercially introduced after this program was well underway. Industrial grade CSPE has greater chemical resistance than the general purpose potable grade.

Elasticized Polyolefin (ELPO)--

Elasticized polyolefin (ELPO) is a blend of rubbery and crystalline polyolefins. This polymeric material was introduced in 1975 as a black unvulcanized, thermoplastic liner, which is heat sealable with a specially designed heat welder for use both in the field and in the factory. ELPO has a low density (0.92) and is relatively resistant to weathering, alkalies, and acids (Haxo et al, 1977). Sheeting of ELPO is manufactured by blow extrusion and is supplied unreinforced, 20-mil thick, 20-ft wide, and up to 200-ft long. It can be fabricated into panels in the factory or shipped in rolls to a site for assembly on the field.

This material was selected as a primary liner in the test program because of its good aging, and moisture and chemical resistance. The specific sheeting used in this study was of 22-mil thickness. It was also studied in immersion, tub, rack, and pouch tests.

Ethylene Propylene Rubber (EPDM)--

EPDM, which was first produced commercially in 1962, is a terpolymer of ethylene, propylene, and a diene monomer that introduces a small number of double bonds into the polymer molecule which are sites for vulcanization of the rubber. The unsaturation is in the side chains of the polymer and not in the main chain, as in the case of butyl rubber. Such a location of unsaturation imparts good ozone, chemical, and aging resistance to the rubber.

> Note: EPDM is chemically similar to, and can be covulcanized with, butyl rubber; consequently, it is now added to butyl rubber liner compounds to improve the resistance to oxidation, ozone, and weathering (Matrecon, 1983).

As it is a wholly hydrocarbon rubber like butyl, EPDM when properly compounded has excellent resistance to water absorption and permeation but has relatively poor resistance to hydrocarbons. EPDM sheeting is available in 54-in. widths and 20- to 60-mil thickness, in both unsupported and fabric-reinforced versions. Special attention is required in seaming crosslinked sheeting because low-temperature vulcanizing adhesives generally need to be used. Sheetings have also been manufactured with unvulcanized EPDM compounds. These are thermoplastic and can be seamed with solvent adhesives and by thermal methods.

The EPDM sheeting exposed in the primary cell was a crosslinked unreinforced membrane of 30-mil thickness. It was also subjected to roof exposure. In addition, three other different EPDM sheetings were subjected to immersion and weather exposure testing; two of these were crosslinked compositions and one was a thermoplastic version which was reinforced with a polyester fabric of 8 x 8 epi thread count.

Neoprene (CR)--

Neoprene is the generic name of a family of synthetic rubbers based upon chloroprene. Neoprene was first introduced in 1932 and thus was one of the first commercial synthetic rubbers. These rubbers are vulcanizable, usually with metal oxides, e.g. MgO, ZnO, and PbO, but also with sulfur. They closely parallel natural rubber in mechanical properties, e.g., flexibility and strength. However, neoprene compositions are generally superior to natural rubber compositions in their resistance to oils, weathering, ozone, and ultraviolet radiation. Most neoprene sheetings are relatively resistant to puncture, abrasion, and mechanical damage. Neoprene membranes have been used primarily for the containment of wastewater and other liquids containing traces of hydrocarbons. They also give satisfactory service with certain combinations of oils and acids (Lee, 1974). Neoprene membrane liners are vulcanized; therefore, special attention is required in seaming because vulcanizing cements and adhesives generally need to be used.

The neoprene exposed as a liner specimen in the primary cell was an unreinforced, crosslinked sheeting of a nominal thickness of 31.25 mils. Two additional neoprene sheetings were exposed in this study in immersion and water tests. All were vulcanized and had no fabric reinforcement.

Polybutylene (PB)--

Polybutylene is a high molecular weight polymer synthesized from butene-1. It was introduced commercially in the United States in the early 1970's and found its major use in plastic pipe. The version of polybutylene used in this project was specifically designed for blown film extrusion. Films of this material feature good flexibility, heat sealability, low moisture vapor transmission, and good creep resistance.

Polybutylene is available in thin films, principally for use as packaging. It has not been manufactured for use as a liner material although there is current interest in it for this use. Polybutylene film was successfully used in making leachate collection bags to use in the study of liners in contact with MSW leachate (Haxo et al, 1982).

Polybutylene was not selected as a primary liner in the exposure cells. It was tested in pouches and used as leachate collection bags. The film first used in this study was unpigmented, i.e. contained no carbon black, and had a thickness of 8 mils. Later, the bags and pouches were fabricated from black-pigmented polybutylene which had a thickness of 6 mils.

Polyester Elastomer (PEL)--

Polyester elastomers form a family of polyether esters which are semicrystalline and thermoplastic. They were introduced commercially in 1972 and feature oil, fuel, and chemical resistance. An experimental sheeting based on this type of polymer was available in 1975 and, because it had oil and chemical resistance, it was selected as a primary material for study and exposure in this program. This sheeting was unreinforced and 7-mils thickness, which made it the thinnest sheeting in the program.

Note: New versions, both of the polymer and of the sheeting, are now available. The sheetings are thicker and reported to be more durable than the one we used in this study.

Polyethylene (PE)--

Polyethylenes are thermoplastic crystalline polymers based upon ethylene. They are currently made in three major types:

1. Low-density polyethylene (LDPE).
2. Linear low-density polyethylene (LLDPE).
3. High-density polyethylene (HDPE).

The properties of a polyethylene are largely dependent upon molecular weight, crystallinity, and density. Of the three types, high-density polyethylene polymers exhibit the greatest resistance to oils, solvents, and permeation by water vapor and gases. Unpigmented clear polyethylene degrades readily on outdoor exposure, but the addition of 2 to 3% carbon black can produce ultraviolet protection. Polyethylenes, as generally used, are free of additives such as plasticizers and fillers.

LDPE and HDPE types of polyethylene have been used as liners. Non-fabric-reinforced membranes of low-density polyethylene have been used for 25 to 30 years (Hickey, 1969) in lining canals and ponds. The low-density polyethylene (LDPE) available in thin sheeting tends to be difficult to handle and to field seam. Also, it is easily punctured under impact such as when rocks are dropped on the lining; however, it has good puncture resistance when buried. Linings of high-density polyethylene (HDPE), which were introduced in the USA after this project was underway, are available in sheetings of 20- to 120-mils thickness; special seaming equipment has been developed for making seams of these sheetings both in the factory and in the field. This type of liner is stiff compared to most of the other membranes described. A lining material based on linear-low-density polyethylene (LLDPE), a recently developed version of polyethylene, was introduced in 1982 in 20- and 30-mil thicknesses.

At the time this project was initiated, only LDPE sheeting had been used in the USA as a lining material, and it was available as a thin film at a thickness of about 10 mils. In the study of lining materials for municipal solid waste landfills (Haxo et al, 1982), we found this film to be deficient in puncture, crease, and fold resistances and not satisfactory for impounding hazardous wastes in spite of its good chemical resistance. Consequently, LDPE was not selected for exposure as a primary liner. However, it was tested in pouches, and unpigmented sheeting of 31-mil thickness in immersion tests.

Though HDPE lining material was not available in the USA at the time this project was initiated, an unpigmented HDPE sheeting of 32-mil thickness was tested in immersion tests because of the known chemical resistance of this type of polymer. Neither of the polyethylenes that were tested were specifically commercial lining materials.

Polypropylene (PP)--

Polypropylene is a partially crystalline thermoplastic polymer based on propylene. It is quite hard, stiff, has good chemical resistance, and has potential as a membrane liner as indicated by its recent commercial introduction in Europe.

The specific polypropylene sheeting assessed in this program was an unpigmented, 33-mil sheeting which was immersion tested in 13 wastes or liquid test media. It was not tested as a primary liner.

Polyvinyl Chloride (PVC)--

Polyvinyl chloride is produced by any of several polymerization processes from vinyl chloride monomer (VCM). It is a versatile thermoplastic, which is compounded with plasticizers and other modifiers to produce sheetings having a wide range of physical properties. Polymeric membranes based upon PVC are the most widely used flexible liners.

PVC sheeting is produced in roll form in various widths up to 96-in. and thicknesses of 20 to 30 mils. Most liners are used as unreinforced sheeting, but fabric reinforcement has been used. PVC compounds contain 25% to 35% of one or more plasticizers to make the sheeting flexible and rubber-like. They also contain 1% to 5% of a chemical stabilizer and various amounts of other additives; PVC compounds should not contain any water soluble ingredients. A wide choice of plasticizers can be used in PVC sheeting, depending upon the application and service conditions under which the PVC compound is to be used. Both monomeric plasticizers, such as phthalate esters, and polymeric plasticizers, such as resins, are used with PVC. Plasticizer loss during service is a source of PVC liner deterioration. There are three basic mechanisms for plasticizer loss: volatilization, extraction, and microbiological attack. The use of the proper plasticizers and an effective biocide can virtually eliminate microbiological attack and minimize volatility and extraction. The PVC polymer, itself, is generally not affected by service in buried conditions. It is affected, however, by exposure to ultraviolet light.

PVC sheetings can deteriorate relatively quickly on exposure to weather due to wind, sunlight, and heat, which cause polymer degradation and loss of plasticizer; consequently, when used as liners they are generally covered with soil. Plasticized PVC sheetings are quite resistant to puncture and relatively easy to seam by "welding" with solvents or bodied solvents, adhesives, and heat.

The PVC membrane selected for testing in the primary cells was an unreinforced sheeting of 33-mil thickness. It was tested in six different wastes in the primary cells and in 12 wastes in immersion. Seven additional PVC sheetings were exposure tested in immersion and pouch tests. All were unreinforced and ranged in thickness from 11 to 33 mils. All eight were also tested in the project on liners for MSW leachate (Haxo et al, 1982). In order to furnish tanks for immersion testing, two cells were lined with an unreinforced sheeting of 20-mil thickness.

Testing of Polymeric Membranes

The testing of a polymeric membrane liner before exposure characterizes the specific sheeting and furnishes a baseline for assessing the effects of exposure. Measurement of the same properties during or after exposure to a given environment can be a measure of the effect of the exposure. For many plastic and rubber products there are limits in the changes that can take place in various properties beyond which the product is not serviceable or is likely to fail. At the time this project was started and even continuing to the present time (1983), no such criteria exist for membrane liners. Establishment of such criteria must await results of field performance which will require extended exposure of membranes in service. Measurement of the changes in properties in laboratory and field exposures will yield information as to the compatibility of the liner and the waste liquid which can be useful in selecting liner materials that will perform satisfactorily in a given waste environment. It is important, therefore, to choose the important properties to follow in an exposure study.

Polymeric membranes are tested by a variety of methods which depend upon the type of membrane liner. Sheetings used as liners have been developed by three different industries, i.e. rubber, plastics, and textile. Each has developed standard test methods. Some test methods for one type of sheeting are not appropriate for other types; for instance, using a dumbbell with a 1/4-in. restricted area, such as is used in the testing of rubber vulcanizates, is not as satisfactory for measuring for specification purposes the tensile properties of fabric-reinforced materials as a 1-in. wide strip or a grab tensile specimen.

Note: As we were assessing the effects of exposure on the barrier coating of the membrane, the dumbbell was used and appeared satisfactory because the thread count was not greater than 10 x 10 epi. This is discussed in Section 6.

The testing of materials, particularly with reference to polymeric membranes for the lining of waste disposal facilities, is described by Haxo (1981) and Haxo, et al (1983).

From the viewpoint of testing, there are four basically different types of polymeric membranes:

- Membranes based on crosslinked elastomers without fabric reinforcement.

- Membranes based on thermoplastics or uncrosslinked polymers without fabric reinforcement.

- Membranes based on fabric-reinforced sheetings which can be based upon either crosslinked or thermoplastic polymers.

- Membranes based on semicrystalline polymers without fabric reinforcement.

The methods used for testing polymeric lining materials can be grouped into four categories:

- Analyses to assess composition and to fingerprint the membrane.
- Tests of physical properties, including information regarding construction and dimensions of the membrane.
- Tests to determine properties in stress environments and aging tests in specific exposures or compatibility tests.
- Tests of durability of lining materials under conditions that simulate actual field service.

These tests can include measurements of the following properties:

- Analytical properties before and after exposure to different environments:
    - Ash.
    - Extractables.
    - Specific gravity.
    - Volatiles.
- Physical properties before and after exposure to different environments:
    - Dimensional stability of immersion specimens (before and after).
    - Hardness (before and after).
    - Modulus of elasticity of crystalline membranes (before and after exposure).
    - Ply adhesion in fabric-reinforced sheeting (before and after exposure).
    - Puncture resistance (before and after exposure).
    - Seam strength of factory and field-prepared seams (before and after).
    - Tear resistance (before and after, except fabric-reinforced sheeting.
    - Tensile properties (before and after exposure).
    - Thickness (before and after exposure).
    - Water vapor transmission (unexposed).

The measurement of these properties represents a body of tests which can be used to assess the effects on a liner of an environmental exposure. A change in one property during an exposure is usually accompanied by changes in other properties, but at this stage in liner testing no one property or combination of properties has been correlated with liner performance in the field. In the subsequent subsections, we discuss these properties and how they are tested.

Prior to exposure to the wastes, all of the polymeric membranes were subjected to the tests listed in Table 15. Results of these tests are shown in Appendixes F and G. They served as reference points to measure retention of properties on exposure. Because of the small size of the slab specimens exposed to wastes, weather, etc, the tests performed after exposure were modified and reduced in number of replicates. These tests and the number of replicates are also listed in Table 15.

TABLE 15. TESTING OF POLYMERIC MEMBRANE LINERS

Test	Unexposed sheeting	Exposed sheeting
Analytical properties		
Specific gravity, ASTM D297, D792	Yes	No
Volatiles, Appendix L	Yes	Yes
Ash, ASTM D297, Section 35	Yes	No
Extractables, Appendix M	Yes	Yes
Physical properties		
Thickness	Yes	Yes
Tensile strength and elongation at break[a], ASTM D412	5 in each direction	3 in each direction
Hardness, ASTM D2240, 5 sec Duro A and Duro D if Duro A >80	5 measurements	5 measurements
Tear strength[b], ASTM D624, Die C	5 specimens in each direction	3 specimens in each direction,
Puncture resistance, FTMS 101B, Method 2065	5 specimens	2 specimens
Seam strength, in peel and in shear, ASTM D413	3 tests in each mode	2 tests in each mode
Water absorption or extraction at room temperature and 70°C, ASTM D570	3 at each temperature	No
Water vapor permeability, ASTM E96	3 specimens	No

[a] Properties using special dumbbell.

[b] This test was used initially in testing fabric-reinforced sheeting, as well as unreinforced sheeting, but was dropped in the test of the fabric-reinforced sheeting.

Analytical Properties of Polymeric Membrane Liners--

Because of the wide range of compositions and constructions of flexible polymeric membrane liners that were available at the time this project was initiated and were developed during the course of the project, analysis and fingerprinting of the membranes were needed. Analysis of a polymeric membrane liner furnishes information to:

1. Characterize and identify the specific sheeting.
2. Use as a baseline for monitoring the effects of exposure on the liner.
3. Assess the aggressive ingredients in the waste liquids that were absorbed by the polymeric membrane liners to determine the compatibility of the liner with the waste liquid.

During exposure to waste liquids, polymeric liners may change in composition in various ways that may affect their performance and result in actual failures. Polymeric materials may absorb water, organic solvents and chemicals, organometallic materials, and possibly some inorganics if the liners become highly swollen. On the other hand, the extractable components of the original liner compound may be leached out and result in stiffening and even brittleness on the part of the liner membrane. The solid constituents of a polymeric compound (which include carbon black, inorganic fillers, and some of the curing agents) will be retained in the liner compound, as will the polymer of which the liner is made (particularly if the polymer is crosslinked). If organic materials are similar to the liner in solubility and hydrogen bonding characteristics, the liner may swell excessively. The volatiles and extractables were determined as part of the testing procedure for measuring the effects of exposure.

The following subsections describe the analytical tests performed on the polymeric membranes in this study.

Ash--The ash content of a liner material is the inorganic fraction that remains after a devolatilized sample is thoroughly burned at 550±25°C. The ash consists of (1) the inorganic materials that have been used as fillers and curatives in the polymeric coating compound, and (2) ash residues in the polymer. Different liner manufacturers formulate their compounds differently and determining the ash content can be a way to "fingerprint" a polymeric liner compound.

The residue obtained by ashing can be retained for other analyses (such as metals content) needed for further identification and for providing a reference point to determine trace metals that may have been absorbed by the liner. The test method described in ASTM D297, "Fillers, Referee Ash Method," Section 34, is generally followed in performing this analysis. Ash contents of unexposed membrane liners were determined in accordance with this method. The results for unexposed membranes are presented in Appendix F.

Exposed membranes were ashed in the early stages of the project to assess the amount of inorganics that might have been absorbed. This analysis was dropped because increases were very small when based upon the polymer content of the compound.

Extractables--The extractables content of a polymeric sheeting is the fraction of the compound that can be extracted from a devolatilized sample of the liner with a solvent that neither decomposes nor dissolves the polymer. Extractables consist of plasticizers, oils, or other solvent-soluble constituents that impart or help maintain specific properties such as flexibility and processibility. A measurement of extractables content can be used as part of the fingerprinting of a sheeting. More recently the extracts from liners have been analyzed (Haxo, 1983).

During exposure to a waste, the extractable constituents in a liner may be removed, which may result in property changes. At the same time, the liner may absorb nonvolatilizable constituents from the waste. Measuring the extractable content of unexposed lining materials is therefore useful for monitoring the effects of exposure. The extract and the extracted liner obtained by this procedure can be subjected to further analytical testing, e.g. gas chromatography, infrared spectroscopy, thermogravimetry, etc, for fingerprinting of the liner.

The procedure for extraction generally follows ASTM D3421, "Extraction and Analysis of Plasticizer Mixtures from Vinyl Chloride Plastics," and ASTM D297, "Rubber Products-Chemical Analysis," Paragraphs 16-18. Because of the wide differences among the polymers used in liner manufacture, a variety of extracting media must be used. The method followed in this work is presented in Appendix M which indicates the media used for the extraction of the membranes of each polymer type.

During exposure, the extractable content of the original materials can increase or decrease depending upon the waste and the length of exposure (Haxo, 1983). In performing an analysis for extractables, the volatiles were removed first. Since some of the extractables may have been somewhat volatile, care was taken to make sure that the volatile extractables were not lost in the analysis. As indicated above, the extractables can be analyzed to determine the organic species that may have been absorbed during exposure. These analyses, however, were not performed in this project.

Specific Gravity--Specific gravity is an important property of a membrane liner material which is comparatively easy to determine and can give an indication of the composition and identification of a polymeric compound. Specific gravity of exposed specimens can be measured only after the specimen has been devolatilized. However, to make this determination, care must be taken to thoroughly air-dry the specimen before placing it in the oven at 105°C for final drying because dissolved or occluded moisture can form steam and voids in the compound. ASTM Method D792, Method A, and D297, Paragraph 15, are generally used in measuring specific gravity. In this study, we found that specific gravity values of exposed materials were erratic and not sufficiently informative; consequently, we discontinued this test.

Volatiles--The volatile fraction is represented by the weight lost by an unexposed specimen of the liner on heating in a circulating air-oven at 105°C for two hours. Polymeric compositions generally contain a small amount of volatiles (<1.0%), usually moisture. The specimen used in this study was a disk cut from the membrane.

The test for volatiles was also used to determine the direction of the grain that had been introduced in the membrane during manufacture. By identifying the orientation of the 2-in. disk specimen with respect to the sheeting at the time the specimen was died out, the grain direction can be identified. The grain direction must be known so that tensile and tear properties can be determined in machine (grain) and transverse directions. Upon heating in the oven at 105°C, most sheetings with a grain will shrink more in the grain direction than in the transverse direction.

Volatiles need to be removed before determining ash, extractables, and specific gravity. Ash and extractables are reported on a dry basis (db). Volatiles contents of the unexposed membrane liners are presented in Appendix F. Monomeric plasticizers that are generally used in PVC compositions have a limited volatility and can slowly volatilize at 105°C. Thus, the air-oven test must be limited to two hours. The procedure used for the determining the volatiles of the unexposed membranes is presented in Appendix L.

After exposure to a waste, a membrane generally increases in volatile content which, in most of our work, was made up primarily of water but also can contain volatile organics. In this project, no attempt was made to differentiate between these two volatiles. In our recent work, however, the volatiles due to moisture and those due to organics are identified (Haxo, 1983).

As performed in this project, the volatiles were removed by air-drying the disk specimen at room temperature for a week, after which the temperature was raised to 50°C and held for 20 hours. The specimen was then heated for two hours at 105°C. Most of the volatiles data on exposed materials were obtained by this method.

Physical Properties of Polymeric Membrane Liners--

Tensile properties--Tensile properties of polymeric sheetings are measured in tension with a stress-strain tester. The properties that are measured depend on the type of polymeric sheeting and include the following:

- Tensile at fabric break (if fabric-reinforced).
- Elongation at fabric break (if fabric-reinforced).
- Tensile at yield (if semicrystalline).
- Elongation at yield (if semicrystalline).
- Tensile at break of sheeting.
- Elongation at break of sheeting.
- Stress at 100% and 200% elongations.

Tensile testing is probably the most widely used test method in the rubber and plastics industries for testing polymer compositions and products. It must be recognized that, even for a given polymeric material, the values for the tensile properties vary with speed of test, i.e. the rate of jaw separation, specimen size, direction of test with respect to the "grain" in the sheeting, temperature, and humidity. "Grain" is introduced in a sheeting during the processes of calendering or extrusion in the manufacture of the sheeting.

Changes in tensile properties can be used to monitor the effects of exposure on a lining material. In many rubber and plastics applications, a 50% loss in tensile strength or in elongation, or a 50% increase or decrease in modulus, indicates that the product has become unserviceable. These criteria are probably not applicable to liners; nevertheless, major changes in properties after a relatively short-term exposure indicate the incompatibility of a liner and a waste.

At the time the project was initiated, we were interested in exploring the effects of exposure on properties on an equal basis. It was decided to test the tensile properties of all of the materials in accordance with the same test method so that comparisons could be made directly without having to make allowances for differences that would arise from the use of different procedures. ASTM Method D412 was selected because of the shape and size of the Type IV dumbbell and the applicability of the somewhat smaller special dumbbell. The size of the dumbbell allowed for the required number of specimens needed to test the exposed lining material samples in contrast to the ASTM D751 grab tensile specimen size required for fabric-reinforced membranes. The shape of the dumbbell ensures that failure will occur within a certain test area in contrast to strip specimens pulled in accordance with ASTM D882, which tend to break in the jaws. Thus, given the exploratory nature of our study, using a dumbbell-type test specimen and a test speed of 20 inches-per-minute were the most appropriate for determining changes in tensile properties during exposure.

In the later stages of the project, when crystalline sheetings came to be of interest, limited testing was performed at 2 ipm because of the sensitivity of these materials to speed of test.

Note: The test procedures followed currently in determining tensile properties for the different types of sheetings are:

Thermoplastic polymers	ASTM D882 ASTM D638
Crosslinked rubbers	ASTM D412
Semicrystalline polymers	ASTM D638
Fabric-reinforced sheetings	ASTM D751, Methods A and B
Selvage edge of fabric-reinforced sheeting	ASTM D882, D638, or D412, depending on the type of polymer coating compound

Modulus of elasticity--The modulus of elasticity is a measure of the stiffness or rigidity of stiff materials such as HDPE; it is defined as the ratio of stress to corresponding strain in the part of the stress strain curve that behaves according to Hooke's law, i.e. the stress values are linear with respect to the strain. It is also known as Young's modulus. ASTM D883, Method A, is followed in determining the modulus of elasticity of semicrystalline sheetings. This test was used only with semicrystalline materials in the later stages of this project when HDPE and polypropylene membranes became of interest. Data on modulus of elasticity of the unexposed semicrystalline materials are reported in Appendix F.

Hardness--Hardness, in terms of the standard tests for hardness of polymeric materials, is the ability of a material to resist indentation by a small probe of specified shape and dimensions. Although no simple relationship exists between hardness as determined by indentation and other measured properties, it is related to Young's modulus (ASTM D1415-81). Hardness is an easily measured indicator of a quality of a polymeric material and can be used to monitor change in a material. ASTM D2240 is generally used to measure this property; the A scale is used for soft, rubbery materials and the D scale is used for hard materials, i.e. materials that are harder than 80 measured on the A scale. Both instanteous- and 5-second readings are taken, but most of the hardness data reported are 5-second readings.

Tear resistance--Tear resistance is a measurement of the force required to tear a specimen with or without a controlled flaw. The measurement serves as an indication of the quality of the compound and of the mechanical strength of a sheeting, particularly to the type of stresses often encountered during liner installation. The magnitude of the tear resistance value is sensitive to the rate of test and the shape and size of the test specimen.

As with the tensile testing, it was decided that all sheetings should be tested in accordance with the same test method, which in this case was ASTM D624 with Die C. It was recognized that this test method was inappropriate for testing fabric-reinforced sheetings. It was used in the initial testing of all sheetings but was later dropped from the testing of the fabric-reinforced liners. This subject is discussed further in Sections 6 and 7. All sheetings were tested with a crosshead speed of 20 ipm until semicrystalline sheetings came to be of interest. Additional testing of these later materials was then performed at 2 ipm.

Note: Coated fabrics are normally tested in accordance with ASTM D751. Most fabric-reinforced membrane liners are a special type of coated fabric because of the low thread count of the fabric and the low adhesion of the coating to the fabric. Because of the low adhesion between the fabric and the polymer coating, when testing in strict accordance with ASTM D751 the fabric pulls out of the polymer matrix resulting in the threads bundling at the tip of the tear and yielding excessively high test values.

As a consequence, the liner industry currently uses a larger test specimen to test tear resistance than that called for in the ASTM method, i.e. 8 x 8 in. instead of 4 x 8 in.

Puncture resistance--Puncture resistance is a measurement of the force required to force a standard probe of a standard configuration through a membrane at a given rate. The measurement serves to indicate the ability of a material to withstand puncture from above, e.g. equipment, foot traffic, deer hooves, etc and from below, e.g. by irregularities in the substrate, etc. We have used puncture resistance to measure the effects of an exposure on a sheeting; however, there is no universally accepted method for measuring puncture resistance. Nevertheless, two Federal Test Method Standards have found use in assessing puncture resistance of sheetings:

1. FTMS 101B, Method 2031, in which the force to slowly puncture a sheeting by a tetrahedral probe is measured. The specimen is a 10 x 4 in. strip.
2. FTMS 101B, Method 2065, in which the force required to puncture a sheeting with a tapered probe having an end of 0.25-in. diameter is measured.

Because of the small size of the exposure test specimen, we used the method described in Federal Test Method Standard 101B, Method 2065, for measuring puncture resistance, particularly of unreinforced sheetings. The usefulness of this test for fabric-reinforced flexible membrane is limited because of the openness of the weaves normally used. It does not measure the resistance to puncture of a liner by a sharp object falling on the sheeting during installation. However, considering the exploratory nature of the project and since the size of the specimen required for the puncture test with the tetrahedral point (FTMS 101B, Method 2031) was too large in relation to the exposure sample, it was decided that the fabric-reinforced materials would also be tested in accordance with FTMS 101B, Method 2065.

Water vapor transmission--To measure water vapor transmission, ASTM E96 Method BW was used. In this test, a cup with a membrane specimen cover is placed in an inverted position in a controlled temperature, humidity, and air stream. Loss in weight of water from the cup is observed as a function of time. This test is intended for those applications in which one side is wetted under conditions where the hydraulic head is relatively unimportant and the moisture transfer is governed by water vapor diffusion forces. The driving force is supplied by the difference in the relative humidity on the two sides of the membrane. Moisture vapor transmission results are reported in Section 9.

Water absorption--Absorption of water can have adverse effects on many polymeric compositions. Since most waste fluids contain water, the effects of immersion in water on lining materials need to be determined. The effects are monitored in terms of either change in weight, change in dimensions, or both. A water absorption test is included to provide a relatively precise comparative index of all the sheetings in the test. Extended immersion periods are recommended. The test specimens are large enough to die out a tensile dumbbell or to pull as a strip to get an indication of the effect of water absorption on tensile properties. Water absorption tests at elevated temperatures accelerate the effects of immersion in water. We performed water absorption tests on selected sheetings at room temperature (23°C) and at 70°C in accordance with ASTM D570 and the results are presented in Table 16. These and other results indicated that water absorption tests run at 70°C were too severe to serve as accelerated aging tests for most materials. We now use a temperature of 50°C for such testing.

> Note: Resistance to environmental stress-cracking--This is a property that was not used in this work but is of importance with semicrystalline polymeric materials and should be considered in assessing them. Under certain conditions of stress and exposure to soaps, oils, detergents, or other surface-active agents, certain grades of plastics, e.g. polyethylene, may fail by cracking at stress levels below tensile strength. Proper selection of the polyethylene or the addition of one of a variety of rubbery polymers to the HDPE can eliminate this deficiency in polyethylenes. ASTM D1693 can be run to indicate the susceptibility of a polyethylene sheeting to environmental stress-cracking. In this test, specimens having a controlled imperfection are bent and exposed to designated surface-active agents. Failure comes with breaking of the specimen. A stress-crack is defined as an external or thermal crack in a plastic caused by tensile stresses less than its short-time mechanical strength.

Testing of Seam Strength of Factory and Field Systems--

A critical factor in the functioning and durability of a polymeric membrane liner in service is the seam strength between assembled panels of the sheeting. Testing of seams was performed to ensure that the method of seaming a particular material is adequate.

Seams were tested in shear and peel modes. Elevated temperatures are often used in seam strength tests. The peel test of seam strength is significantly more sensitive to the effects of aging and exposure than is shear testing.

Seam strength in shear was measured in accordance with ASTM D882, Method A, modified so as to allow for the testing of 1-in. wide strips containing a seam. The use of this method arises from ASTM D3083, the PVC liner standard, which recommends that shear tests be performed in accordance with ASTM D882, which is a strip tensile test. The National Sanitation Foundation and others are using this method. Seam strength in the peel mode was measured in accordance with ASTM D413, Machine Method Type B, in 90° peel.

TABLE 16. WATER ABSORPTION OF PRIMARY POLYMERIC MEMBRANE LINER MATERIALS AT ROOM TEMPERATURE AND AT 70°C[a]

		Water absorbed, % by weight											
		At room temperature						At 70°C					
Polymer	Liner number[b]	1 week	11 weeks	44 weeks	100 weeks	159 weeks	186 weeks	1 week	11 weeks	44 weeks	100 weeks	159 weeks	186 weeks
Butyl rubber	57R	0.82	3.22	4.50	6.4	10.92	10.76	4.62	17.54	53.9	103.2	c	141.3
Chlorinated polyethylene	77	1.63	5.53	10.2	12.5	16.85	16.51	15.9	58.4	140.0	179.3	178.89	196.14
Chlorosulfonated polyethylene	6R	3.44	6.97	10.9	16.3	20.81	19.42	22.1	131.0	245.6	370.5	38.16	471.40
Elasticized polyolefin	36	0.39	0.52	0.0	4.5	0.93	0.38	0.36	0.45	0.57	8.7	-4.19	-8.48
Ethylene propylene rubber	26	1.20	1.84	1.49	2.56	3.85	3.61	1.44	4.52	11.20	17.4	21.08	22.60
Neoprene	43R	3.80	13.62	37.8	75.1	67.20	63.11	14.1	107.0	240.0	d	d	d
Polyester elastomer	75	1.07	1.05	0.67	1.31	1.61	0.36	1.28	1.10	0.72	0.22	0.79	d
Polyvinyl chloride	59	1.59	2.34	2.43	2.98	4.53	1.55	4.87	8.25	24.0	25.5[e]	26.17[e]	26.86[e]

[a] ASTM D570-63 specimens (1 x 3 inches) in deionized water.

[b] R indicates that liner was fabric reinforced.

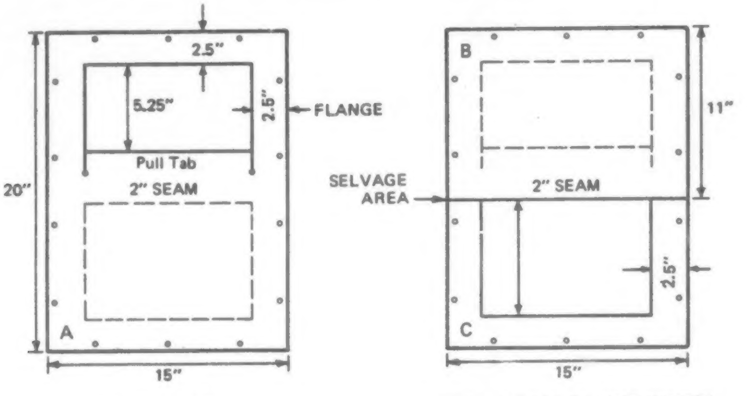
[c] Specimens not weighed.

[d] Specimens disintegrated.

[e] Specimens became hard, indicating loss of plasticizer.

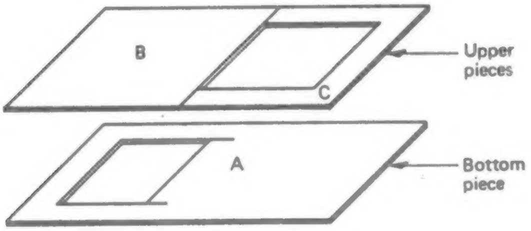
Fabrication and Mounting of Membrane Liner Specimens in Cells

Test specimens of the flexible membranes to place in the cells for long-term exposure were fabricated as shown in Figure 8. Each test specimen featured a seam that could be tested both in shear and peel modes. Each seam was made according to the instructions of the specific membrane supplier and, in some cases, with his materials. The seams were 2-in. wide and were parallel to the machine direction of the sheeting as is usually encountered in the seaming of sheets. In the case of fabric-reinforced membranes, the top piece, "B", incorporated in the seam a selvage edge which faced the waste, thus avoiding exposing the cut ends of the reinforcing fabric directly to the waste liquid. The underside of the liner has a 1.5-in. tab left free for peel testing.



BOTTOM PIECE

UPPER PIECES FACING WASTE



ISOMETRIC DRAWING
(Sketch not to scale)

Figure 8. Test specimens for long-term exposure of membranes in primary cells. Pieces A and B are seamed together with a 1.5-in. strip of polyethylene along the edge behind the peel tab. Piece C is butted against the seam edge and tacked in place as a spacer to produce a double thickness around the cell flange area.

Prior to fabricating the liner specimens to place in the long-term exposure cells, sample seams were made and tested. If the seams were satisfactory, fabrication of the specimens began. Seams were considered satisfactory if, when tested in shear, they did not fail in the adhesive. In the case of vulcanized liner materials, the seams of which intrinsically fail in the adhesive, the seams were considered satisfactory when they reached strength levels previously determined to be acceptable. If the seams were unsatisfactory, a second set of sample seams was made and tested. In some cases, cleaning of the liner surface prior to seaming had been inadequate; in others, insufficient adhesive had been applied.

Figure 9 shows an unassembled exposure cell, with a membrane liner specimen, prior to assembly (Haxo et al, 1977). Table 17 presents results of the testing of seams in unexposed liner specimens.

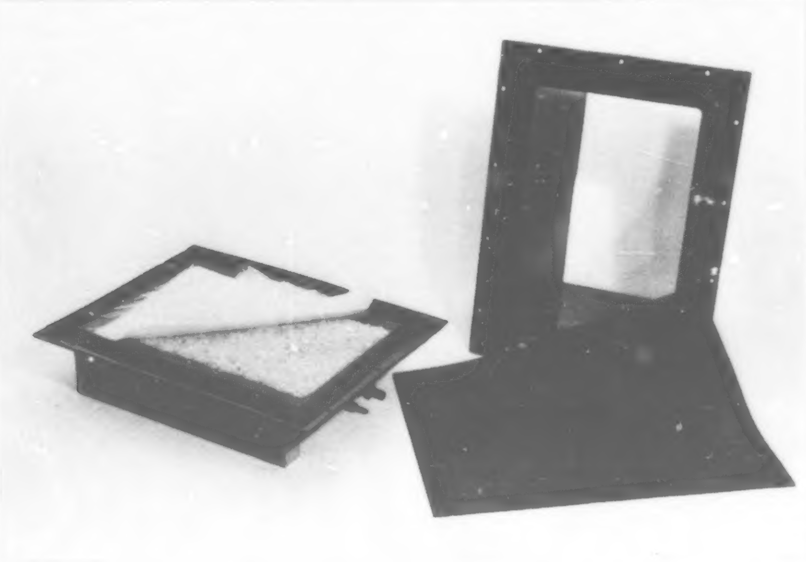


Figure 9. Unassembled exposure cell used for membrane liner specimen. Shown are the tank and the base filled with silica gravel and a membrane liner specimen (Haxo et al, 1977).

The polymeric membrane specimens, with the exception of neoprene, were mounted in the cells with a sealing caulk applied to the upper surface of the specimen facing the flange of the tank. A gasket of 0.25-in. closed-cell neoprene sponge was placed between the liner specimen and the flange of the base. The sponge gasket compensates for irregularities in the two flanges and allows for a better seal. The neoprene liners were mounted with the same 0.25-in. neoprene sponge on the upper side of the liner and no caulk was used.

TABLE 17. ORIGINAL STRENGTH OF SEAMS IN MEMBRANE LINER SPECIMENS MOUNTED IN PRIMARY CELLS

Item	Test method	Polymeric membranes, Matrecon serial numbers							
		Butyl 57	CPE 77	CSPE 6R	ELPO 36	EPDM 26	Neoprene 43R	Polyester elastomer 75	PVC 59
Method of bonding		Cement LTV[a]	Solvent	Cement	Heat	Cement LTV[a]	Cement LTV[a]	Heat	Solvent
Width of lap seam, in.		2	2	2	0.5	2	2	0.5	2
Time after seaming, days		39	234	40	b	293	39	b	39
Peel strength (avg)[c], ppi	ASTM 1876 (Modified)	8.0	21.4	23.2	...	4.9	8.2	...	15.2
Peel strength (max), ppi Locus of failure[d]		8.8 AD	22.4 AD	28.5 AD+DEL	21.0 SE	5.2 AD	9.1 AD-LS	20.8 SE	16.0 AD
Shear strength[e] (max), ppi Locus of failure[d]	ASTM D882	>84.8 CL	48.3 SE	62.1 SE	28.8 SE	39 AD	45.7 AD-LS	>21.2 CL	>69.1 CL
% of tensile strength of liner[f]		>119	71	120	49	54	92	>45	>89

[a] LTV = Low temperature vulcanizing or curing.

[b] Seams made by supplier.

[c] Strip specimen 1-in. wide; initial distance between jaws, 2 in.; rate of separation of jaws, 2 ipm.

[d] Locus of failure:
CL = Break at clamp.
SE = Break at seam edge.
AD = Failure in adhesive.
AD-LS = Failure between adhesive and liner surface.
DEL = Delamination in liner.

[e] Strip specimen 1-in. wide; initial distance between jaws, 4 in.; rate of separation of jaws, 2 ipm.

[f] $\frac{\text{Shear value measured at 2 ipm}}{\text{Force to break liner (from tensile strength test at 20 ipm)}} \times 100.$

Two caulking compounds were used in sealing the liners. Since the caulks were exposed to the waste, they were chosen on the basis of preliminary screening tests (Section 6). We used a butyl caulk with the acid and caustic wastes and a polysulfide caulk with the other wastes.

After allowing time for the caulk to cure, the cells were tested by filling with water. A couple of the cells leaked slightly, but could be sealed by tightening bolts in the cell flanges to compensate for the compression set taken by the neoprene foam. One liner had to be completely remounted. Another cell that passed the water test and was filled with waste developed a slight seepage between the liner and the exposure cell tank. This liner eventually had to be remounted.

SECTION 6

EXPOSURE IN PRIMARY CELLS

INTRODUCTION

The principal effort of this project was to assess experimentally a range of lining materials under conditions that simulate the service of liners in place in waste storage and impoundment facilities. The objectives of this part of the project were to expose a sufficiently large sample of each selected liner material to different wastes, to measure the effects of the exposures upon properties, and to measure the quantity and quality of the waste that passes through the liner, particularly in the case of soils and admixes. The results were to be used to assess the general performance of the selected lining materials in contact with actual wastes and to determine their durability. Ultimately, it is desired to be able to estimate service lives of lining materials under different conditions.

In this section the design and construction of the test cells in which specimens of the lining materials were exposed are described as are the preliminary compatibility test procedures which led to the selection of combinations of waste streams and lining materials for the long-term exposure testing. The monitoring of the test cells during exposure is described with particular emphasis on the soil and admixed lining materials. The recovery of the liner materials after usually two exposure periods is described and unusual effects are noted. Finally, for each of the liner types, the results of exposure are presented and discussed. A complete tabulation of the exposures of the liner specimens in the different wastes is presented in Appendix A.

DESIGN AND CONSTRUCTION OF PRIMARY EXPOSURE CELLS AND ANCILLARY EQUIPMENT

Primary Exposure Cells

Each individual exposure cell was designed to perform as a permeameter and to be adaptable to liner specimens ranging from 20 mils to 12 in. in thickness as shown schematically in Figure 10, for membranes and thick liner specimens. The cell provides for test specimens 10 x 15 in. in area and a volume of waste of approximately 1 cu ft at a depth of 1 ft. Photographs of the exposure cells for thick admix specimens and for membrane liners are shown in Figures 9 and 11.

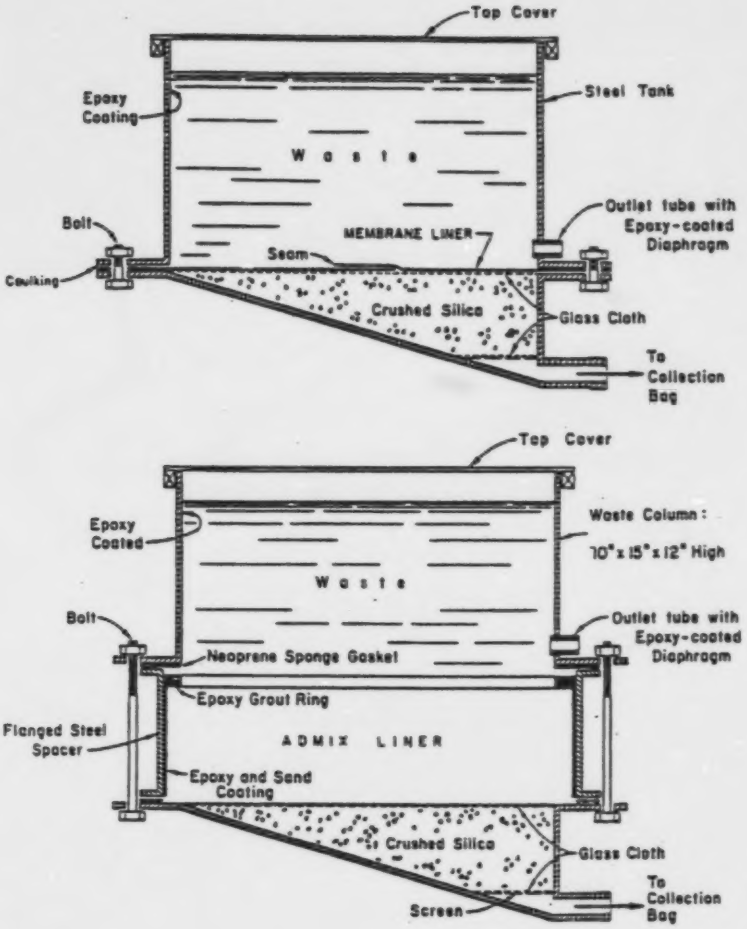

Top Cover
Epoxy Coating
Steel Tank
Waste
Bolt
Outlet tube with Epoxy-coated Diaphragm
MEMBRANE LINER
Seam
Caulking
Crushed Silica
Glass Cloth
To Collection Bag
Top Cover
Epoxy Coated
Waste Column: 10" x 15" x 12" High
Waste
Bolt
Outlet tube with Epoxy-coated Diaphragm
Neoprene Sponge Gasket
Epoxy Grout Ring
Flanged Steel Spacer
ADMIX LINER
Epoxy and Sand Coating
Crushed Silica
Glass Cloth
To Collection Bag
Screen

Figure 10. Design of cells for long-term exposure of membrane liners (top) and soil and admix liners (bottom) to the different hazardous wastes. A liner specimen is 10 x 15 in. in area.

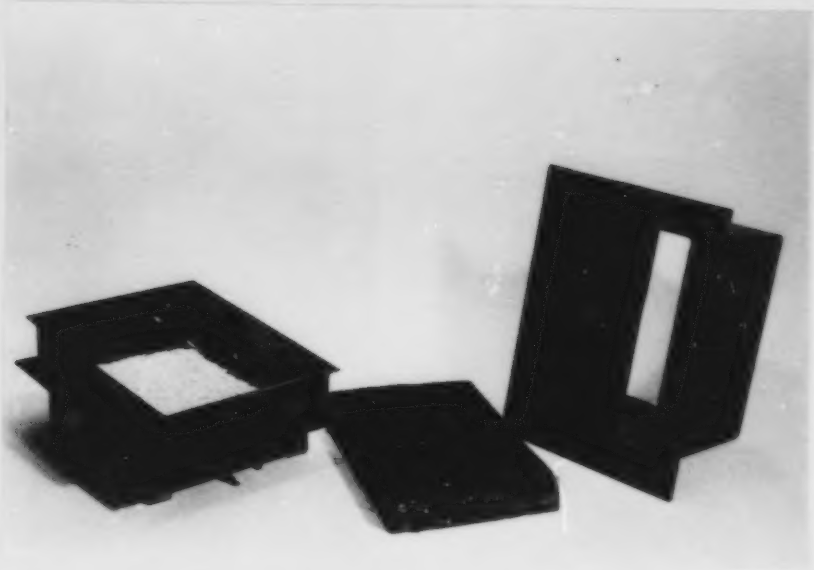


Figure 11. Unassembled exposure cell used for thick admix specimens. Shown are the three components, the tank, the spacer for the thick liners, and the base loaded with silica gravel. Shown also is an hydraulic asphalt concrete specimen.

One hundred forty-four cells were fabricated from 11-gauge steel, coated on the inside with a chemically resistant epoxy coating, Epoxy 3, and on the outside with a rust-preventive paint. (See Appendix H for details regarding materials used in constructing cells and mounting liner specimens.)

After the first 24 cells were coated with paint brush and pad, it was decided that spraying would be a more economical way to coat the remaining 120 cells. A jig was constructed to facilitate the coating of the cell tank interiors. The cell bases were laid out next to each other on long tables so that 20 could be sprayed as a group.

The spraying of the primer presented no problem as its viscosity was within the medium range for paint. Spraying of the two-part epoxy coating, Epoxy 2, was somewhat more difficult; a quart of the mixed coating has a potlife of 42 minutes at 77°F and a viscosity of 30 poise, the consistency of a very heavy paint.

One coat of the primer was applied, followed by a coat of the two-part epoxy coating, Epoxy 2. A second coat of Epoxy 2 was applied along the bottom weld on the tank sections. All coatings were tested for pinholes with a spark tester.

The bases of the cells were finished similarly to the tanks. They were filled with a chemically-inert high quality crushed silica in order to prevent reactions and contamination of the waste which might seep through the liner.

Spacers for mounting the admix liners were fabricated of different heights, depending on the thickness of the liner. For those liners compacted in-place, i.e. the soil-cement, native soil, and the modified bentonite, the inside surfaces were coated first with the primer and Epoxy 2, and then with a second coat of Epoxy 2 sprinkled with sandblasting sand. The rough wall surface was formed to reduce or eliminate a wall effect which might result in the waste by-passing the liner. A ring seal of epoxy-sand was cast around the periphery of soil, the admix, and the sprayed-on liner specimens to increase path length of the waste percolating through the liner and to reduce further the possibility of wall effects and channeling. The rings were cast of Epoxy 1 and Epoxy 2 filled with sandblasting sand (Appendix H).

Lids for each of the 144 cells were fabricated of hardboard, wood, and polyethylene, and installed on the filled cells. These lids reduced water loss by evaporation and prevented accidental contact with the hazardous waste by people or animals; they were not intended to be either water or air-tight.

Rack for Holding Exposure Cells

The exposure cells were mounted on a rack constructed outdoors on a concrete slab at the Richmond Field Station of the University of California, Berkeley. This rack consisted of 6 sections, each of which held 24 exposure cells. Each section had 2 tiers and was 4 x 8 x 3 ft. These 6 sections were arranged in 2 rows of 3 sections each, which were placed 4 ft apart, as shown in Figures 12 and 13. A corrugated plastic cover was constructed over the rack at a height of 8 ft to protect the cells from rain and sunlight. The design of the racks afforded good access to the cells; each could be removed or replaced with relative ease.

Although the cells were outdoors, they were well protected from the heat of the sun. The temperature was relatively uniform, generally in the range of 50 to 60°F, and did not drop below freezing.

Silica Gravel

The gravel used to fill the cell bases and support the liners was a crushed quartz from the Bear River (California); it was selected because of its high silica content (99%) and the low content of constituents soluble in acid which might contaminate the seepage through the liner specimens. The seepage was to be collected and characterized to assess the attenuation by and time for waste to percolate through the liner. The nominal size was 3 to 6 mm (0.125 to 0.25 in.); individual pieces of stone were as long as 25 mm in one dimension, since the gravel had been crushed in a jaw crusher which tends to produce a high percentage of long and flat particles. The gravel was thoroughly washed and dried before placing in the bases.

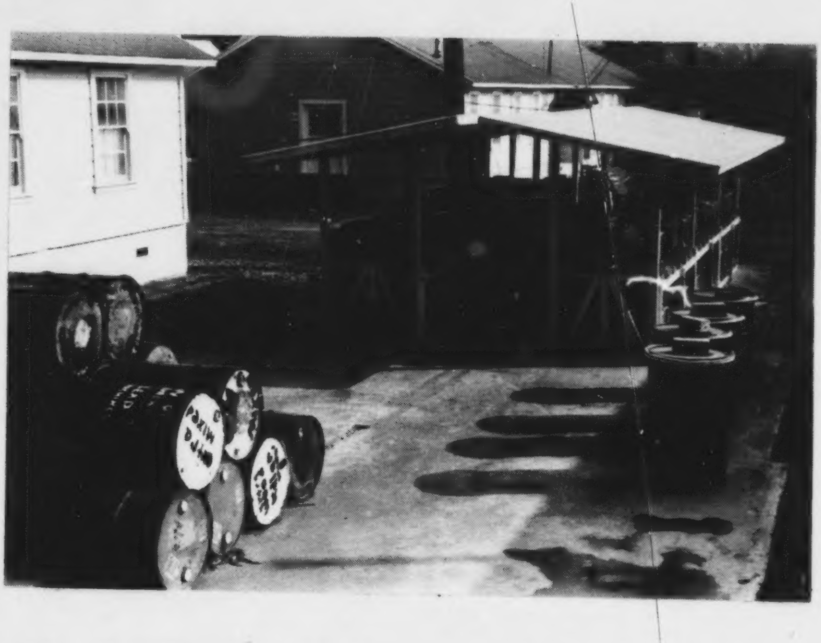


Figure 12. Overall view of the rack holding the exposure cells.

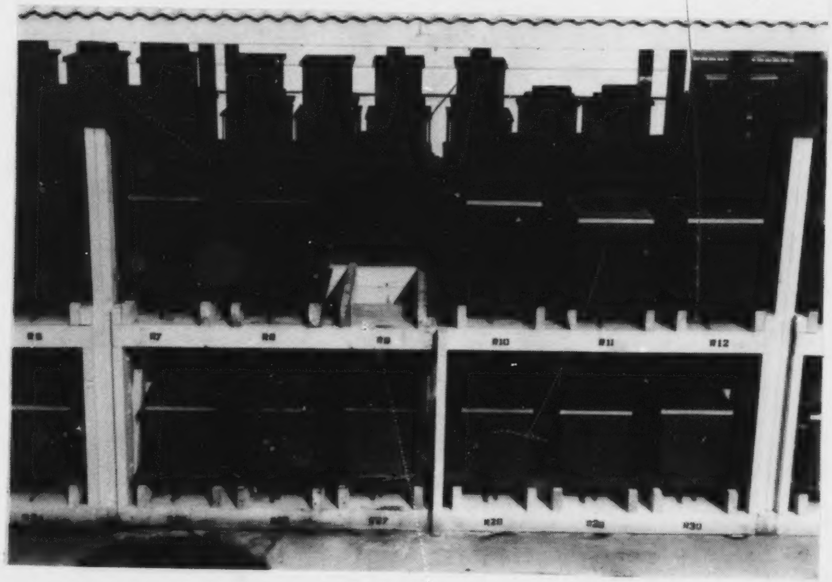


Figure 13. View of cells on the rack. These include cells containing membrane and admix liners.

SOILS

Selection of Wastes for Exposure to Soil Liner

A preliminary series of tests of the compatibility of the Mare Island soil was run with the different wastes that had been obtained. The objective of this testing was to determine which wastes should be placed in the long term exposure test as it was desired to include only those combinations of liners and wastes that would not fail in a short time. The lead, oily, and pesticide wastes were considered compatible with the soil and, therefore, were not included in the compatibility test.

The compatibility test that was used for the soil is described by Haxo et al, 1977. Basically, it consisted of sealing 2-in. diameter cylindrical specimens in one end of 8-in. long, 54-mm i.d. Pyrex cylinders with Epoxy 1 and Epoxy 4 (Appendix H). Three days after the epoxy cement was cast, the cylinders were filled with the wastes to a depth of 5 inches.

The fine-grain soil specimens were prepared by molding the soil at a water content of 20 parts per 100 parts dry soil in a 2-in. diameter mold. The soil was compacted in 5 lifts with a high-frequency impact tool followed by double-end static compaction. The height of the specimens was about 2 inches.

The specimen in acidic waste, "HNO_3-HF-HOAc", released a few bubbles immediately and continued to release bubbles slowly for about a week. Within one day the surface of the specimen developed a flaky appearance and the epoxy peeled off the glass in both "HNO_3-HF-HOAc" and "Spent caustic"; within 2 days the same effect was noted with the specimen in "Slop water."

When probed with a glass rod after 7 days of exposure, the specimen exposed to "Slop water" had a hard surface; the specimen exposed to "Spent caustic" was soft on the surface, and the surface of the specimen exposed to "HNO_3-HF-HOAc" was flaky. There was no leakage from any of the specimens. After 11 days of exposure, a slightly damp spot appeared at the center of the bottom of the specimen exposed to "Spent caustic." After 16 days the damp spot remained unchanged and was neutral to litmus paper.

Because of the bubbling and the flaky appearance of the soil, it was decided to delete the acid waste "HNO_3-HF-HOAc" from the primary exposure test. The wastes included in the primary exposure test of the soil liner and the number of cells into which each was loaded are shown in Table 18.

Monitoring the Cells

The monitoring of the cells consisted of:

1. Measuring the amount of liquid seeping through the liner. Data were kept for incremental collections as well as cumulative collections as a function of time.

2. Measuring the level of the wastes in the tanks periodically for comparison with the seepage.

3. Determining the quality of the seepage effluent to assess the flow of waste through the liner. The measurements included pH, electrical conductivity, total solids, and, periodically, elemental analysis.

TABLE 18. WASTES INCLUDED IN PRIMARY EXPOSURE TESTS OF THE SOIL LINER

Identification of waste		Matrecon serial number	Number of cells
Type	Name		
Alkaline	"Spent caustic"	W-2	2
Lead	...	W-14	2
Oily	"Oil Pond 104"	W-5	2
	"Slurry oil"	W-15	2
Pesticide	"Weed killer"	W-11	2

A polybutylene bag was attached to the base of each of the cells containing soil liners into which the seepage could continuously drain freely. The polybutylene was an effective bag for this purpose as it formed a durable seam. However, these bags had to be protected from the light as the polybutylene was unpigmented. There were some losses of effluent due to failure of bags because of UV degradation. The failed bags were replaced with bags made of black polybutylene when it became available.

The measurement of the composition of the effluent was important so that changes in the waste that percolated through the liner would be observed. Typical test results of the seepage are presented in Tables 19 and 20 and Figure 14 for two cells with soil liners and "Spent caustic" and pesticide wastes. The results on these two cells are discussed below in the subsection "Discussion of Soil Liner Results."

Dismantling of Cells and Testing of Soil Specimens

Four cells with soil liners were dismantled for testing the soils, particularly with reference to the metals distribution in the soil following exposure to the different wastes. The three cells containing "Spent caustic" waste, lead waste, and "Oil Pond 104" waste had been in exposure for 958 days, and a cell containing "Slurry oil" waste had been in exposure for 696 days. The wastes were removed from the tanks and then the spacers with the soil liners were removed from the cell assembly.

The soil was sampled either by cutting narrow slits in the soil and removing blocks of the liner material or by forcing a brass tube into the liner, avoiding compaction, and noting original depths and locations. The

TABLE 19. MONITORING OF CELLS - COLLECTION AND ANALYSIS OF SEEPAGE FROM CELL WITH MARE ISLAND SOIL LINER AND SPENT CAUSTIC WASTE (W-2)

Date	Time, days		Amount, mL		Total dissolved solids, %	pH	Waste level below tank top, in.
	Incremental	Elapsed	Incremental	Cumulative			
3-5-76	...	...	Cell assembled				
4-3-76	0	0	Cell filled				
11-15-76	223	223	282	282	21.13	7.2	...
2-1-77	78	301	472	754	20.01	7.0	...
6-23-77	142	443	860	1614	21.87	6.2	...
11-19-77	149	592	500	2114	23.24	7.2	2.1
5-10-78	172	764	635	2749	...	6.0	2.4
9-15-78	128	892	480	3229	...	...	2.6
4-27-79	224	1116	724	3953	23.13	6.4	3.1
2-4-80	Collection lost - bag replaced						
4-21-80	77[a]	1476	253	4206[b]	...	4.6	4.1
9-15-80	147	1623	17[c]	4223[b]	...	3.2	...
11-7-80	53	1676	206	4429[b]	20.43	6.9	8.0
2-6-81	91	1767	208	4637[b]	19.83	7.1	7.9
5-7-81	90	1857	303	4940[b]	22.00	6.5	5.4
8-7-81	92	1949	285	5225[b]	...	6.7	6.5
11-6-81	91	2040	248	5473[b]	...	6.5	6.8
2-8-82	94	2134	180	5653[b]	...	6.7	6.7
5-7-82	88	2222	240	5893[b]	20.95	6.8	7.1
8-6-82	91	2313	260	6153[b]	...	6.7	7.3
11-10-82	96	2409	195	6348[b]	21.60	6.0	7.6
2-8-83	90	2499	255	6603[b]	...	6.6	7.7
5-6-83	87	2586	197	6800[b]	...	6.5	8.0

[a] Data for April 21, 1980, represents 77 days accumulated seepage. A ruptured bag prevented accumulation of seepage between April 27, 1979, and February 4, 1980.

[b] Cumulative amount based on all available data but does not include estimates of lost collections between April 23, 1979, and September 15, 1980.

[c] Most of collection lost.

TABLE 20. MONITORING OF CELLS - COLLECTION AND ANALYSIS OF SEEPAGE FROM CELL WITH MARE ISLAND SOIL LINER AND WASTE PESTICIDE (W-11)

Date	Time, days		Amount, mL		Total dissolved solids, %	pH	Waste level below tank top, in.
	Incremental	Elapsed	Incremental	Cumulative			
2-23-76	...	...	Cell assembled				
3-1-76	0	0	Cell filled				
11-15-76	259	259	316	316	19.48	7.0	...
2-1-77	78	337	292	608	19.01	7.2	...
6-23-77	142	479	680	1288	18.13	6.8	...
11-19-77	149	628	665	1953	16.75	7.4	4.8
5-10-78	172	800	710	2663	15.42	6.6	5.0
9-15-78	128	929	500	3163	...	...	5.8
4-23-79	220	1148	1035	4198	13.56	7.4	6.6
2-4-80	Collection lost - bag replaced						
4-21-80	77[a]	1512	22	4220[b]	...	7.5	8.1
9-15-80	147	1659	20[c]	4240[b]	...	3.5	...
11-7-80	53	1712	193	4443[b]	9.59	5.6	4.3
2-6-81	91	1803	295	4728[b]	9.22	7.4	3.8
5-7-81	90	1893	325	5053[b]	9.00	6.7	9.3
8-7-81	92	1985	283	5336[b]	...	7.1	9.6
11-6-81	91	2076	320	5656[b]	8.92	6.8	10.0
2-8-82	94	2170	251	5907[b]	...	7.0	10.1
5-7-82	88	2258	315	6222[b]	8.89	7.2	10.4
8-6-82	91	2349	330	6552[b]	...	7.3	10.7
11-10-82	96	2445	266	6818[b]	8.72	6.9	11.1
2-8-83	90	2535	166	6984[b]	...	7.5	11.4
5-6-83	87	2622	207	7191[b]	...	7.5	11.7

[a] Data for April 21, 1980, represents 77 days accumulated seepage. A ruptured bag prevented accumulation of seepage between April 23, 1979, and February 4, 1980.

[b] Cumulative amount based on all available data but does not include estimates of lost collections between April 23, 1979, and September 15, 1980.

[c] Most of collection lost.

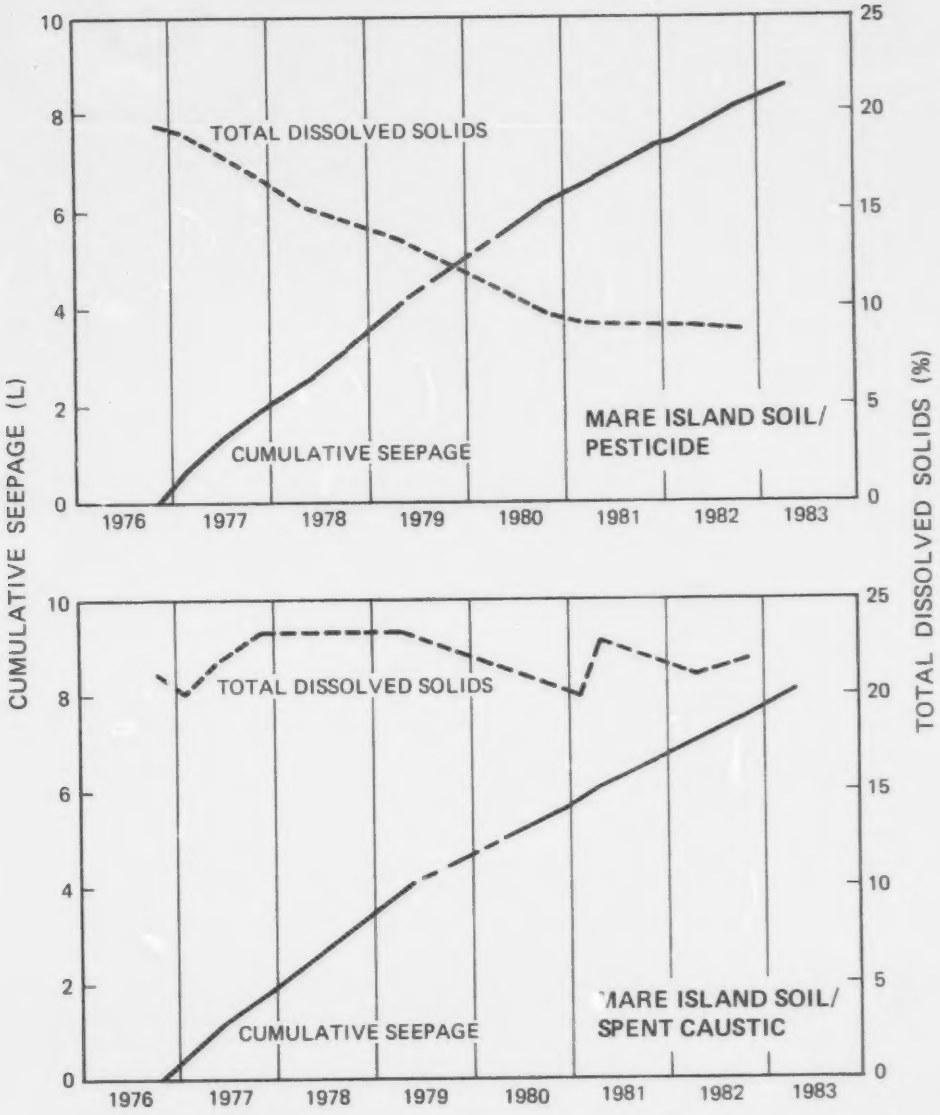


Figure 14. Monitoring of cells with Mare Island soil liner. The cumulative seepage and the total dissolved solids of the seepage are shown for the cell containing pesticide waste, which has a total dissolved solids content of about 0.4%, and for the cell containing the "Spent caustic" waste, which has a total dissolved solids content of approximately 22%. Adjustments were made for the losses of seepage during the period between April 23, 1979 and September 15, 1980.

samples, along with samples of exposed bentonite-sand mixtures and soil cement, were then submitted to H.E. Doner of the University of California, Berkeley for analysis of selected trace metals and study of metal migration in the soil.

Movement of Waste Constituents in the Soil Liners

Soil samples were analyzed for six chemical species by H.E. Doner. The blocks or corings of soil were trimmed to remove possible contamination on the surface. Samples were then taken by cutting sections at various depths of the cores. The sampled sections were air-dried, ground, and sieved through a 2 mm stainless steel screen and a subsample taken to determine moisture content. Extraction of Cd, Cr, Cu, Ni, and Pb was accomplished by digestion of 5.0 g soil in 30 mL 4N HNO_3 at 70°C for 16 hours (Ganje and Page, 1974) followed by the determination using a flame atomic absorption spectrophotometer equipped with a background corrector. Mercury was extracted from a sample 0.1 g by a mixture of $KMnO_4$, aqua regia, and $K_2S_2O_8$ and analyzed by a mercury vaporization method with an atomic absorption spectrophotometer. The analytical data for six chemical species in three exposures ("Spent caustic," lead waste, and "Slurry oil") are presented in Table 21. The last line of the table shows the background concentration present in the unexposed soil. The distribution of cadmium, chromium, copper, lead, mercury, and nickel in the soil liner after prolonged exposure to "Oil Pond 104" waste is shown in Figure 15.

TABLE 21. ELEMENTAL ANALYSIS AVERAGED OVER ALL DEPTHS FOR MARE ISLAND SOIL

	Metal concentration, µg/g oven-dried soil					
Treatment	Cadmium	Chromium	Copper	Mercury	Nickel	Lead
"Spent caustic"	0.37	61.9	65.4	0.34	79.9	30.2
Lead waste	0.43	60.6	70.7	0.31	89.8	a
"Slurry oil"	0.39	59.9	70.3	0.37	89.3	30.9
Unexposed soil	0.29	60.8	65.0	0.32	88.9	28.1

[a] See Figure 16.

The fact that the distribution of different chemical species on the profile was uniform, and that the average concentration values for each of the liners are close to the values for the unexposed soil, is a reflection of the low concentration of the chemical species in the respective wastes. This is shown to be the case for the "Spent caustic" and lead wastes in Table 22. Table 22 also indicates that, unlike the lead and the "Spent caustic" wastes, the "Oil Pond 104" waste is quite concentrated with regard to the investigated chemical species. As a consequence, during the 31.5 months over which the flow was monitored, a clear profile was obtained for all elements as shown in Figure 15. The concentrations of lead and

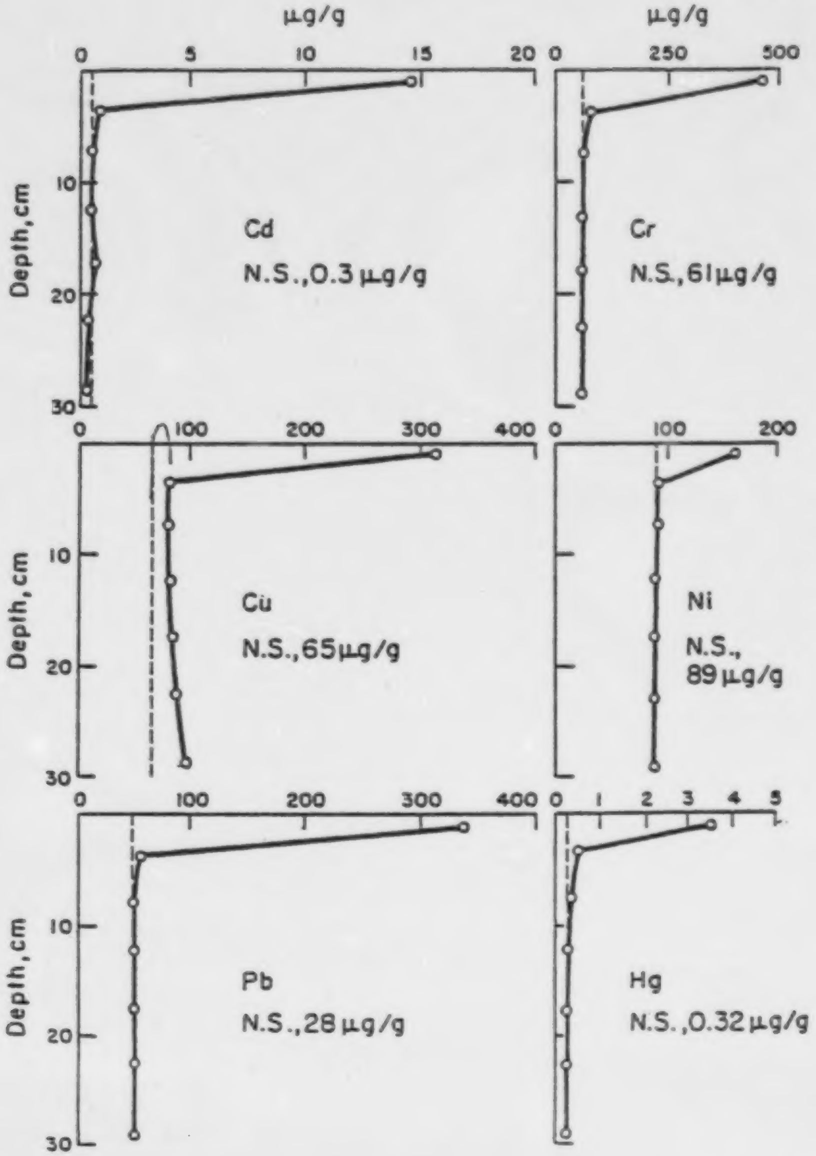


Figure 15. Distribution of cadmium, chromium, copper, lead, mercury, and nickel in soil liner after 31.5 months (958 days) exposure to "Oil Pond 104" waste.

TABLE 22. CONCENTRATION OF SIX CHEMICAL SPECIES IN THREE WASTES[a]

Chemical species	Waste					
	Lead		"Spent caustic"		"Oil Pond 104"	
	$\mu g\ mL^{-1}$	N	$\mu g\ mL^{-1}$	N	$\mu g\ mL^{-1}$	N
Cadmium	0.76	1.4×10^{-5}	0.6	1.1×10^{-5}	8.0	1.4×10^{-4}
Chromium	1.9	...	0.8	...	290.0	...
Copper	1.1	3.5×10^{-5}	2.3	7.2×10^{-5}	33.0	1.0×10^{-3}
Lead	95.0	9.2×10^{-4}	5.0	4.8×10^{-5}	170.0	1.6×10^{-3}
Mercury	0.06	6.0×10^{-7}	0.003	3.0×10^{-8}	1.6	1.6×10^{-5}
Nickel	0.90	3.1×10^{-5}	1.8	6.1×10^{-5}	4.0	1.4×10^{-4}

[a] See Table 3 for more details.

chromium are high in the "Oil Pond 104" waste; thus, these elements could compete with major cations for adsorptive sites. The enrichment of lead in the very top layer of the soil exposed to lead waste is shown in Figure 16.

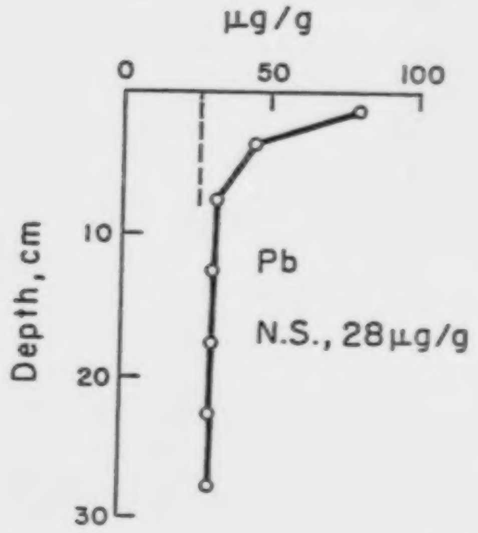


Figure 16. Distribution of lead in soil liner after 31.5 months (958 days) of exposure to lead waste.

Discussion of Soil Liner Results

Analysis indicated that the lead waste was a solution of 10^{-3} N concentration with respect to lead, and this concentration was the main reason for the sharp profile that is shown in Figure 16. This figure indicates that, on the average, the concentration of lead in the contaminated soil was 80 $\mu g\ g^{-1}$ at 0-2 cm and 44 $\mu g\ g^{-1}$ at 2-5 cm. Taking into account the lead concentration of 28 $\mu g\ g^{-1}$ in the unexposed soil, one finds a concentration difference of 52 $\mu g\ g^{-1}$ for the first two centimeters and 16 $\mu g\ g^{-1}$ for the next three centimeters of liner. Assuming that all lead is divalent, these values correspond to 0.05 and 0.015 meq/100 g soil, which is a level of adsorption below the equilibrium capacity of a solution of 1.6×10^{-3} N concentration. Thus, one can say that when the Mare Island soil was exposed to a waste with a high concentration of lead, it retained lead and consequently exposed the underlying soil layer to a smaller concentration of lead.

Figure 15 shows, in the case of "Oil Pond 104" waste, a retention of all six chemical species in the first two centimeters of the soil liners. Figure 15 also shows that, with the possible exception of copper, the contamination with the five heavy metals was superficially localized, i.e in the top two centimeters of the soil.

Analytical data concerning the concentration of the six chemical species in "Oil Pond 104" waste (Column 2) is presented in Table 23.

TABLE 23. CONTAMINATION OF MARE ISLAND SOIL WITH SELECTED CHEMICAL SPECIES WHEN EXPOSED TO "OIL POND 104" WASTE

Chemical species (1)	C_0[a] $\mu g\ mL^{-1}$ (2)	(mN) (3)	Δc[b] ($\mu g\ g^{-1}$) (4)	M_1[c] (μg) (5)	c_a[d] ($\mu g\ g^{-1}$) (6)	(meq/100 g) (7)
Cadmium	8	0.142	14	3.1	10.9	0.0194
Chromium	290	11.15	380	113.7	266.3	1.0242
Copper	33	1.039	250	12.9	237.1	0.7463
Nickel	4	0.136	70	1.6	68.4	0.2330
Lead	170	1.641	300	66.6	233.4	0.2253
Mercury	1.6	0.016	3.1	0.6	2.5	0.0025

[a] C_0 = Concentration in "Oil Pond 104" waste.

[b] Δc = Concentration difference between contaminated and uncontaminated soil.

[c] M_1 = Mass of element present in one pore volume (1 PV) associated with 1 g of soil (1 PV = 0.392 mL), assuming elemental concentration of liquid in pore space is equal to C_0.

[d] c_a = Concentration of species either as precipitated constituent or as adsorbed cation.

Column 3 of Table 23 is calculated assuming that all species are of valence "+2"; this is not true for the chromium. Column 4 is the difference between the "exposed to Oil Pond 104" and unexposed soil and, therefore, is a measure of contamination. Column 5 is the amount of contaminant corresponding to one gram of saturated soil (porosity equal to 0.509), assuming that the concentration of a particular species in the pore liquid is equal to its concentration in "Oil Pond 104" waste. Columns 6 and 7 represent the content of a species associated with the soil solid phase, either as a precipitated constituent or more likely as an adsorbed cation.

Data in Column 3 indicate that the concentrations of cadmium, copper, nickel, and lead vary between 1.36 x 10^{-4} N and 1.64 x 10^{-3} N, which is a range characteristic for major constituents, such as Ca^{2+}, in uncontaminated soils. Thus, one can expect this group of "trace" elements to compete with cations, such as Ca^{2+}, Mg^{2+}, and K^+, for adsorptive sites. Since the cation exchange capacity of Mare Island soil is in excess of 10 meq/100 g soil and since the total milliequivalents (less chromium) in Column 7 is exactly equal to 1 meq, the adsorptive potential of the 0-2 cm layer has not been reached after 2.6 years of exposure.

The abrupt change in concentration at a very shallow depth for all six elements with the "Oil Pond 104" waste, can be explained in several ways. First, the concentration of chemical species in this waste is much higher than in either the lead waste or the spent caustic; thus, the chemical species were able to compete for adsorptive sites successfully. Second, the low flow rate of 10^{-8} cm s^{-1} resulted in a large residence time; thus, cations and soil surfaces were provided sufficient time to interact. Third, "Oil Pond 104" waste, being lighter than water and having low solubility in water, should have yielded a quite abrupt oil-wetting front with a minimal dispersion above and below the front.

Table 23 allows the calculation of the volume of "Oil Pond 104" waste which must have percolated through the 0-2 cm layer to generate the retention indicated in Column 6, assuming that the cation-soil reaction is far from the equilibrium and thus that all the cations in the liquid phase are transferred into the soil matrix. Based on such an assumption, one can calculate for the element cadmium, for instance, that 10.9 μg were adsorbed as a result of the leaching of a gram of soil by 10.9/3.1, i.e. 3.52 PV of "Oil Pond 104" waste. Adding to this value the 1 PV which is in the pore space one obtains 4.52 PV. Since 1 PV corresponding to 1 g soil is equal to 0.392 mL and there are 2348.4 g soil in the first 2 cm of the liner, the total "Oil Pond 104" waste which must have reached the 2 cm depth is equal to 4.16 L. Table 24 presents similar data for the other chemical species, except chromium. The values for cadmium, lead, and mercury are well in the range of the amount of "Oil Pond 104" waste which penetrated the surface of the soil. The values for copper and nickel are several times as large as the actual amount of leachate collected. This apparent anomaly can be explained if one considers that at any time during the flow process the soil surface is an "open" boundary between the oil which is already inside the soil pore space and the oil above the liner; thus there is permanent diffusion of cations from the "source" above ground into the "sink" inside the soil.

TABLE 24. VOLUME OF "OIL POND 104" WASTE REQUIRED TO PERCOLATE THROUGH THE 0-2 CM SOIL LINER TO PRODUCE THE CONTAMINATION INDICATED IN TABLE 23[a]

Element	Volume of "Oil Pond 104" waste required, L
Cadmium	4.16
Copper	17.84
Nickel	40.30
Lead	4.15
Mercury	4.76

[a] In Column 4 of table.

In addition to the chemical considerations presented above, the cells were monitored over a period of 6.5 years for flow rates and general characteristics of the effluent, such as pH, electrical conductivity (EC), and total dissolved solids (TDS). Figure 14 presents the cumulative amounts of effluents and the concentrations of dissolved constituents (TDS) for two representative cells; one cell contained a soil liner and was loaded with pesticide waste and the second cell contained a soil liner and was loaded with spent caustic. Similar graphs were constructed for all cells, the flow behavior of which is analyzed in subsequent paragraphs. This analysis is based on the following:

- Area of the flow boundary between the waste and the soil equal to 903 cm^2.
- Soil volume in the cell equal to 27.53 L.
- Soil bulk density, ρ_B equal to 1.3 g cm^{-3}.
- Soil mass in the cell equal to 35.8 kg.
- Soil porosity, n equal to 0.509.
- Soil effective porosity, n_0 equal to 0.458 (as n_0 assumed to be 0.9 n).
- Waste depth over the soil equal to 30.5 cm.

The results, calculated on the basis of these data and also on the basis of an average TDS for each cell and cumulative infiltration (assumed equal to total effluent) are presented in Table 25.

TABLE 25. AMOUNT OF SALT DISCHARGED IN THE EFFLUENT AND AVERAGE PERMEABILITY VALUES FOR COMPACTED SOIL LINERS

Waste identification[a]	Salt discharged in the effluent, percent, on soil	Effluent, pore volumes	Elasped time[b], days	Average permeability (K), 10^{-8} cm s^{-1}
Pesticide (W-11)	3.6	0.79	2622	2.7
Pesticide (W-11)	3.3	0.67	2622	2.3
Lead waste (W-14)	3.8	0.91	2620	3.1
"Oil Pond 104" (W-5)	3.1	0.60	2611	2.0
"Spent caustic" (W-2)	4.9	0.63	2586	2.2

[a] Matrecon waste serial number in parentheses.

[b] As of May 6, 1983.

The large TDS concentration in the effluents is the result of the large proportion of salts in the original Mare Island soil. Figure 15 shows that, when the waste is pesticide and practically free of dissolved salts, the TDS drops during the tested period to approximately half its original value. This is to be expected if one considers that approximately 0.67 pore volume was leached. At the same time, salt in the Mare Island soil diffused into the pesticide waste to increase the concentration of salt. The original waste had a total solids value of 1.2% and an electrical conductivity of 3,000 µmho. Late in the exposure the supernatant waste had a total solids value of 3.1% and an electrical conductivity of 45,000 µmho.

In the case of the "Spent caustic" waste concentrated in TDS, this fact is reflected by the high concentration of salts in the effluent throughout the whole testing period of 6.5 years.

Table 25 indicates the low permeability of the Mare Island soil. Because less than one pore volume of effluent was collected, the results cannot be interpreted as reflecting the interaction between the Mare Island soil and different wastes, but rather the permeability with regard to the initial soil solution present in the freshly compacted soil. The charts comprising flow data for all cells are very much alike both in terms of the cumulative outflow values and in terms of the constancy of the K value during the testing period for any one of the cells. Both features point to the fact that Mare Island soil is quite insensitive to chemical treatment; however, since only a small amount of waste crossed the waste-soil boundary during the test period, this statement is made with caution. The fact that the soil resisted alterations in flow properties, either as a result of a change in the state of flocculation or because of volume changes or matrix dissolution, is probably related to the very large ratio of nonclay minerals:clay minerals in the fraction of soil smaller than 2 µm. Overall, the Mare Island soil performed quite satisfactorily during the first 6.5 years of exposure to a variety of hazardous wastes.

ADMIXED MATERIALS

Three types of admixed materials were included in this study, i.e. asphalt concrete of hydraulic-grade, bentonite which had been treated with a polymer, and soil-cement. The preparation of these materials is described in Section 5. In this subsection, the compatibility testing and the selection of wastes for the long-term exposure are described, and the results of the testing over periods of up to 6 years are presented.

Asphalt Concrete

Selection of Combinations of Wastes and Liners--

Ten asphalt concrete liner specimens were selected for the exposure test from those cut from the sheet that was originally compacted on a concrete slab. The selection was made on the basis of appearance and uniformity of thickness, i.e. 2.5 in. Because of the sensitivity of the asphalt to hydrocarbons, the oily wastes were not included in the exposure test program of the asphalt concrete liners. The wastes and the number of cells into which they were loaded are:

Acidic waste	- "HNO_3-HF-HOAc"	2
Alkaline waste	- "Spent caustic"	2
Lead waste	-	2
Pesticide	- "Weed killer"	2
Water	- EBMUD[a] tap water	2

---

[a] East Bay Municipal Utility District, California.

The lead waste was included because it appeared to have been almost completely water; however, one component of the blend which made up the lead waste used in the exposure tests did contain a significant amount of hydrocarbon.

Monitoring of Cells--

After the cells were loaded with wastes, some leakage occurred mostly through the seals around the edges of the tanks, but the waste did not seep through the liner. Tightening the bolts around the flange was sufficient in most instances to eliminate further leakage of this type, although it was necessary to remount several of the cells.

Relatively early, seepage occurred through several of the asphalt concrete liners, one from a cell containing a "Spent caustic" waste, one from a cell containing lead waste, and the other two from cells containing the strong acidic waste. It was necessary to remove these cells from the exposure test and dismantle them. In all cases, there were significant leaks through the liners as determined by flooding the liners with water and pressurizing the lower part of the cells and observing the bubbling of air through the asphaltic concrete.

The asphalt concrete liner specimen taken from the cell containing the acidic waste showed a considerable amount of dissolved aggregate at the surface. Presumably, the effect on the aggregate was the result of reaction of the aggregate with the waste which contained some hydrofluoric acid; also, the top layer of the asphalt was quite spongy. This liner specimen was cored to determine its properties and the properties of the asphalt.

An asphaltic liner that had been in the cell containing the "Spent caustic" waste showed leaks; they were all on one-half of the liner as observed by pressurizing the base. We coated the top layer of the liner in the area of the leaks with a seal-coat and returned the cell to the exposure test, this time filling it with the second alkaline waste, "Slop water," which was considerably more alkaline than the first. Only a small amount of leakage occurred in the subsequent six years. No seepage occurred in the liner in the second cell containing the "Spent caustic" waste.

Of the cells still in operation in July, 1983, there has been no seepage in either the second cell containing the lead waste or the remaining cell containing pesticide waste. However, both of the cells containing tap water seeped; total cumulative seepages were 623 mL and 1299 mL, respectively, over 7.5 years.

Dismantling of Cells and Recovery and Testing of Liners--

On removal from the exposure cells, the individual asphalt concrete liner specimens were cored and subjected to the following tests:

- Permeability.
- Voids content.
- Unconfined compressive strength.
- Asphalt viscosity following extraction.

The results of these tests are presented in Table 26.

Acidic waste--The hydraulic asphalt concrete (HAC) liners in the two cells containing the acidic waste failed and were removed and tested after 40 and 199 days of exposure. As indicated above, the surface of both liners was rough and spongy and much of the rock aggregate had dissolved due to reaction with the waste which contained some hydrofluoric acid.

TABLE 26. ASPHALT CONCRETE (HAC) AFTER EXPOSURE TO HAZARDOUS WASTES[a]

Type of waste	...		Acidic				Lead	Pesticide	
Name and number of waste	...		"HNO$_3$-HF-HOAC" (W-9)				(W-14)	"Weed killer" (W-11)	
Cell number	...		C-31		C-26		C-27	C-23	
Exposure time, days	...		40		199		192	569	
HAC concrete[b]									
Coefficient of permeability, cm s^{-1}	1.3×10^{-8}		2.6×10^{-8}		2.4×10^{-9}		...	...	
Density, g/cm^3	2.322		...		2.294		...	2.351	
lb/ft^3	145.0		...		143.2		...	146.8	
Voids content, % volume	4.6		...		5.8		...	3.4	
Compressive strength, psi	268		162		234		21[c]	237	
Retention of original strength, %	...		60		87		8	88	
Extracted asphalt									
Depth of slice from top, cm	0-1	2-3	0.3[d]-1	2-3	0-1	2-3	...	0-1	2-3
Viscosity[e] at 25°C and 0.01 s^{-1}, MP	4.90	1.78	3.66	1.22	f	1.78	2.73	2.55	1.78
Shear susceptibility	0.10	0.70	0.09	0.13	...	0.14	0.11	0.03	0.14
Penetration at 25°C, calculated[g]	48	71	53	87	...	75	60	58	75

[a] The effect on asphalt of exposure to weather at Richmond Field Station for 1,485 days (4.07 years) was measured. Asphalt extracted from top 6 mm (0.25 in.) of weather-exposed piece, cut from the same slab from which liner test specimens were cut, had a viscosity at 25°C and 0.01 s^{-1} shear of 15.8 MP and a calculated penetration at 25°C of 28.

[b] Thickness of liner specimen: 6.3 cm (2.5 in.).

[c] Compressive strength measured using "Pocket Penetrometer" because cores were too soft to hold shape and be tested. The viscosity measured on the recovered asphalt does not represent the very low viscosity of the asphalt binder of the concrete in the in-place liner; the binder contained hydrocarbon absorbed from the waste. This hydrocarbon, along with the extracting solvent, evaporated in the recovery.

[d] Asphalt from 0 to 0.3 cm was too hard to test.

[e] Micro = Caltrans Method 365.

[f] Asphalt from 0-1 cm was too hard to test.

[g] Calculated from absolute viscosity at 25°C.

The unconfined compressive strength of the 2-in. cores taken from the exposed concrete as well as from a piece of the original concrete that had been retained in the laboratory for 16 months are reported in Table 26. The cores cut from liners exposed to acid waste had lost strength significantly in comparison with the laboratory-aged concrete.

The consistency of the asphalt was measured to determine the effect of aging after exposure to the waste. The viscosity of asphalt extracted from different depths of the liner was determined and the results are reported in Table 26.

The asphalt taken from the top surface upon drying was hard and powdery and had such a high viscosity that it could not be measured. The asphalt extracted from the first centimeter of depth was considerably harder than the original material and harder than that taken from deeper in the pavement. On the other hand, it was essentially the same as that of material which had been retained in a can in an uncompacted form or had been exposed to air in the compacted form for about 16 months. It appears that the low permeability to air of asphalt concrete has protected the inner asphalt in the compacted concrete.

Alkaline Waste--The liner in one cell containing the "Spent caustic" waste leaked early. The tank was emptied, filled with water, and the base was pressurized with air. Bubbles rose to indicate the spot of leakage. The small holes were repaired with a coating and the cell was reloaded with "Slopwater" waste and returned to test. Neither liner specimen has been recovered at this time (July 1983) to determine changes in its properties.

Lead Waste--One cell containing lead waste seeped and was dismantled after 192 days of exposure. The asphalt concrete liner from this cell absorbed large amounts of the organic fraction in the waste and became very soft, almost a slush. It could not be lifted from the base without sagging and therefore was slid off of the gravel onto a piece of plywood. The asphalt had softened and some had flowed down into the base and covered the top surface of the white gravel below. Tests with a pocket penetrometer yielded:

Unconfined compressive strength	Location
1.3 kg/cm^2	Between edges of crust
3.7 kg/cm^2	On top of crust
1.5 kg/cm^2	In a soft area

Cores were then cut by hand with 2-in. brass tubing applying only slight pressure and rotation. The cell had to be tipped on its side to remove the cores which fell apart when placed in bottles.

Pesticide waste--The cell with the pesticide waste was removed after 569 days of exposure. The liner remained in satisfactory condition, retaining its compressive strength and showing only slight hardening on aging.

Normal aging of asphalt concrete--Two-inch specimens were also molded of the hydraulic concrete which had been retained in closed cans in uncompacted form. The unconfined compressive strength of these test specimens was probably higher than the original material because of the higher viscosity. The strength of this concrete which was retained after immersion in water was also relatively high and the percentage loss in strength during immersion was not as great as experienced by concrete exposed to the waste (Table 27).

Bentonite-Sand Mixtures

Selection of Combinations of Wastes and Liners--

Ten liner specimens based on bentonite-sand mixtures were prepared. Eight were based on a treated bentonite (Bentonite A) from one manufacturer and two on a treated bentonite (Bentonite B) from a second manufacturer. The cells with liners based on Bentonite B were loaded with the lead waste, "Slurry oil" waste, "Oil Pond 104" waste, and the pesticide waste. The two cells with liners based on Bentonite A were loaded with pesticide waste.

In the compatibility tests that were run to determine the waste liner combinations, the acidic and alkaline wastes broke through the bentonite in a relatively short time. As a consequence, these wastes in combination with the bentonites were not included in the primary exposure test.

Monitoring the Cells--

Of the ten cells with liners of modified bentonite, measurable seepage occurred in seven cells, one of which failed allowing the waste ("Oil Pond 104") to come through the liner. Seepage was collected regularly, measured, and analyzed for solids, pH, and electrical conductivity. The level of waste in the tank was also monitored. Irrespective of the type of waste above the two types of bentonite liners, the quality of the seepage was not greatly different. The seepage in the cells containing the pesticide waste was approximately twice as great for the two Bentonite A liners as for the two Bentonite B liners. Monitoring data for the cell that contained the liner of the Bentonite A-sand mixture and the "weed killer" waste are presented in Table 28. The electrical conductivity data were erratic and did not correlate with the data on total dissolved solids.

Dismantling of Cells--

Two cells with Bentonite B-sand liners were dismantled; one cell contained "Oil Pond 104" waste (after 982 days of exposure) and the other cell contained lead waste (after 986 days of exposure). When the spacers containing the bentonite-sand mixtures were opened and the liners sampled, it was found that there had been considerable "fingering" of the wastes into these liners. The oil wetting front was very irregular and mostly vertical cracks were observed; oil penetrated along these cracks all the

TABLE 27. DENSITY AND COMPRESSIVE STRENGTH OF ASPHALT CONCRETE (HAC)

Sample description	Specific gravity	Voids content, %	Unconfined compressive strength at 25°C	
			psi	Retention of strength, %[a]
Laboratory compacted briquets prepared December 1976 from uncompacted concrete retained in closed can since August 1975. Tested dry.	2.352	3.4	491	...
	2.351	3.2	453	...
	2.256	7.3	513	...
Average	2.320	4.6	486	...
Tested after 24 h immersion in 60°C water.	2.331	4.3	359	74
	2.343	3.8	365	75
	2.347	3.6	356	73
Average	2.340	3.9	360	74
Core cut from sample compacted in August 1975 and retained in lab until December 1976.	2.323	4.6	268	...
Cores cut from liner in cell exposed to strong acid waste for 3 months, retained under water until tested.	...	...	169	63
	...	...	154	57
	...	...	163	61
Average	...	...	162	60

[a] Retentions for briquets tested after immersion in 60°C water bath reported as percent of average strength of briquets tested dry. Retentions for cores tested after acid waste exposure reported as percent of strength of unexposed core.

way to the bottom of the specimen. Upon dismantling one cell and trying to remove the liner specimen as a whole, it collapsed, breaking along these cracks. This is evidence that, even in a wet state, the bentonite did not succeed in providing enough cohesion to the sand mass to withstand its own weight. No channeling of the waste at the walls of the spacers was evident.

TABLE 28. MONITORING OF CELLS - COLLECTION AND ANALYSIS OF SEEPAGE FROM CELL WITH BENTONITE A-SAND MIXTURE AND PESTICIDE WASTE (W-11)

Date	Time, days		Amount, mL		Total dissolved solids, %	pH	Waste level below tank top, in.
	Incremental	Elapsed	Incremental	Cumulative			
3-26-77	0	0	Cell filled				
11-11-77	230	230	900	900	0.27	8.9	...
5-10-78	180	410	610	1510	0.27	8.2	2.6
9-15-78	124	534	460	1970	...	...	2.6
4-27-79	224	758	728	2698	0.31	7.2	3.3
2-4-80	Collection lost - bag replaced						
4-21-80	77[a]	1118	174	2872[b]	...	6.6	4.0
9-15-80	147	1265	111[c]	2983[b]	...	6.4	...
11-7-80	53	1318	260	3243[b]	0.28	6.4	4.5
2-6-81	91	1409	310	3553[b]	0.29	6.6	1.5
5-7-81	90	1499	339	3892[b]	0.30	6.1	4.9
8-7-81	92	1591	372	4264[b]	...	5.9	5.0
11-6-81	90	1681	332	4596[b]	0.29	6.1	5.5
2-8-82	94	1775	200	4796[b]	...	6.1	5.4
5-7-82	90	1865	270	5066[b]	...	6.0	5.7
8-6-82	91	1956	475	5541[b]	0.30	5.8	5.8
11-10-82	96	2052	218	5759[b]	0.30	5.8	6.0
2-8-83	90	2142	0	5759[b]	...	...	6.0
5-3-83	87	2229	380	6139[b]	...	5.9	6.2

[a] Time interval before collection on April 21, 1980 was 77 days; earlier collections between April 27, 1979 and February 4, 1980 were lost.

[b] Cumulative amount based on all available data but does not include estimates of lost collections between April 27, 1979 and September 15, 1980.

[c] Most of collection lost.

This type of liner is probably not satisfactory for these types of waste. The use of a soil cover on the bentonite layer to produce an overburden would probably reduce the fingering effect.

Migration of Constituents--

The Bentonite B-sand liner was sampled in a fashion similar to that used in the sampling of the soil. The samples of the Bentonite B-sand mixture that had been exposed to the lead waste were submitted to H.E. Doner at the University of California, Berkeley, for analysis and trace metals distribution. Results are presented in Table 29.

TABLE 29. ELEMENTAL ANALYSIS AVERAGED OVER ALL DEPTHS FOR BENTONITE B-SAND LINER

Treatment	Metal concentration, μg/g oven-dried soil					
	Cadmium	Chromium	Copper	Mercury	Nickel	Lead
Lead waste	0.19	3.17	7.1	0.008	1.7	9.0

The pH of the Bentonite B-sand mixture was between 8.0 and 8.3 with the higher value in the top 4 cm. This high value may be due to the presence of calcareous material.

Discussion of Results on Modified Bentonite Liners--

Even though there had been some flow through the liner, the distribution of the heavy metals throughout the different depths of the liner was uniform, which indicates that there was no contamination by the waste. Therefore, the data were averaged as shown in Table 29.

Soil-Cement

Selection of Combinations of Wastes and Liners--

The ten liner specimens of 4-in. thick soil-cement were placed in exposure to five wastes, as shown in Table 30.

TABLE 30. WASTES USED IN EXPOSURE TEST OF SOIL-CEMENT

Waste identification		Matercon serial number	Number of cells
Type	Name		
Alkaline waste	"Spent caustic"	(W-2)	2
Lead waste		(W-14)	2
Oily waste	"Slurry oil"	(W-15)	2
	"Oil Pond 104"	(W-5)	2
Pesticide waste	"Weed killer"	(W-11)	2

Due to the known sensitivity of the soil-cement to acid, the acidic waste "HNO_3-HF-HOAc" was not included in the exposure test. This sensitivity was confirmed in compatibility tests (Haxo et al, 1977).

Monitoring of Cells--

During the first year of exposure, no seepage occurred in any of the ten cells containing soil-cement liners. The first five specimens were dismantled for testing after 328 to 527 days of exposure. No seepage through the soil-cement liner specimens in the remaining five cells has occurred in more than six years, except for a small amount (60 mL) in the cell containing "Oil Pond 104" waste.

Effect of Exposure on Properties--

One set of the soil-cement lining materials was recovered after 328 to 625 days of exposure to the five wastes and the individual liners were cored and tested for compressive strength. In all cases, compressive strength of the exposed soil-cements was greater than that of the corresponding unexposed material as shown in Table 31. Some blistering occurred in the epoxy-asphalt coating (Seal C) that was applied to one-half the surface of each specimen.

TABLE 31. COMPRESSIVE STRENGTH OF SOIL-CEMENT LINERS AFTER EXPOSURE TO DIFFERENT WASTES

Exposure	Matrecon waste serial number	Exposure time, days	Compressive strength, psi	Percent of dry unexposed soil-cement
Unexposed[a], tested dry	...	0	1321	...
Unexposed[b]	...	0	604	46
"Weed killer" waste	W-11	ca 560	1592	121
"Spent caustic" waste	W-2	627	1639	124
Lead waste	W-14	627	1363	103
"Oil Pond 104" waste	W-5	627	1798	136
"Slurry oil" waste	W-15	328	1687	128

[a] Stored in plastic bag, but had dried out.

[b] Similar core to (a); tested after immersion in water at room temperature for one week.

Analyses and Trace Metals Distribution--

Cores were also cut from the exposed soil-cement liner specimens for submission to H.E. Doner of the University of California, Berkeley, for determination of the trace metals distribution. The cores were sectioned dry with a diamond saw at three different depths and then treated similarly to the soil samples (see above).

The results of the analyses are presented in Table 32. As noted in the table, the values are similar for all four of the exposed soil-cement liners which indicates that the values equal those for unexposed soil-cement and that there was no contamination by the wastes.

TABLE 32. ELEMENTAL ANALYSIS AVERAGED OVER ALL DEPTHS FOR SOIL-CEMENT

Exposure	Metal concentration, μg/g oven-dried soil					
	Cadmium	Chromium	Copper	Mercury	Nickel	Lead
"Spent caustic" waste	0.02	27.4	27.8	0.16	38.4	22.2
Lead waste	0.02	26.6	28.3	0.16	38.0	20.2
"Oil Pond 104" waste	0.02	26.8	27.5	0.18	37.9	20.7
"Slurry oil" waste	0.02	27.5	28.4	0.17	39.4	21.5

Discussion of Soil-Cement Liner Results--

As evidenced by the lack of seepage, the soil-cement had very low permeability and was quite compatible with the specific wastes to which it was exposed, i.e. the oily, alkaline, and pesticide wastes. The acidic waste had been eliminated in the preliminary compatibility tests.

SPRAYED-ON ASPHALT

Preliminary Compatibility Tests

The membrane based on emulsified asphalt sprayed on a nonwoven fabric was placed under three of the six wastes: pesticide, "Spent caustic," and lead. Two cells with each waste were placed in exposure. The acid waste was excluded because of the severe hardening it caused the asphalt in preliminary exposure tests and the oily wastes were excluded because of the high mutual solubility of the asphalt and such wastes. Later in the program the remaining four cells were loaded with deionized water and with 5% aqueous solution of NaCl for use in immersion testing.

Monitoring of Cells

No seepage of the original six cells occurred up to 489-671 days of exposure when the first group of cells was dismantled to assess the effects of respective exposures on the liners. Subsequently, the second cell with

"Spent caustic" waste was dismantled because of the failure of the cell tank due to corrosion by the salt-caustic mixture. Also, after 780 days of exposure the liner in one of the cells with 5% NaCl solution began to leak and has continued to leak slowly. At the time of the writing of this report (July, 1983) this has amounted to 966 additional days.

Dismantling of Cells and Recovery and Testing of Liners

After 489-671 days of exposure, three cells with pesticide, "Spent caustic," and lead wastes were dismantled and the liners recovered and tested. Two additional cells, one with "spent caustic" waste and the other with lead waste, were dismantled after 1480 and 1348 days, respectively, and the liners recovered and tested. Results are reported in Table 33.

The volatiles content of the liners exposed to the "Spent caustic" waste and the lead waste increased significantly. Presumably, the volatiles were principally water.

A sample of each liner was extracted and the viscosity of the recovered asphalt determined. There is some indication that, for the long exposure, the asphalt hardened after having softened somewhat during the shorter term exposures.

As of May 1983, the remaining five liners were still being exposed to the pesticide waste, deionized water, and NaCl solution. Four were functioning satisfactorily; however, the fifth with 5% NaCl solution is leaking. The electrical conductivity of the effluent is similar to that of the liquid above the liner.

This type of liner appears to be compatible with water and with dilute waste waters but is questionable for use with oily wastes, acid wastes, and possibly other wastes. It definitely requires compatibility testing with the specific waste which is to be impounded.

POLYMERIC MEMBRANES

The exposure testing of polymeric membranes formed the principal part of this project. As indicated in Section 5, a large number of polymeric membranes were candidates for the test. Based upon their properties, the eight membranes listed in Table 34 were selected for the primary exposure test. It had originally been planned to include only nonreinforced membranes in the program but, because of product unavailability at the time the project was started, two fabric-reinforced membranes were substituted. In the planning stage, it was recognized that several of these lining materials would not be compatible with some of the wastes that were available for the test program. Because only a limited number (144) of cells were fabricated for the project and, because it was desired to include only those combinations of liners and wastes that would have reasonable durations of exposure, preliminary compatibility tests were deemed necessary. By eliminating some combinations which were expected to fail, it was possible to test the maximum number of different combinations for the desired periods of exposure.

TABLE 33. EMULSIFIED ASPHALT SPRAYED ON NONWOVEN FABRIC AFTER EXPOSURE TO HAZARDOUS WASTES[a]

Waste type		Alkaline		Lead		Pesticide
Waste name		"Spent caustic"		...		"Weed killer"
Matrecon waste serial number		(W-2)		(W-4)		(W-11)
Exposure time, days	None	671	1480	656	1348	487
Asphaltic liner:						
Volatiles content of liner, %	0.26	12.9	15.3	18.6	21.5	1.45
Water vapor permeability[b], metric perm cm	6.7×10^{-2}	...	...	...	...	...
Extracted asphalt:						
Viscosity at 25°C, P x 106						
at 0.05 s-1	6.1	4.40	8.14	5.52	6.49	5.4
at 0.01 s-1	5.9	4.22	8.03	5.56	6.91	5.4
at 0.001 s-1	5.7	4.00	7.72	5.92	7.53	5.4
Shear susceptibility	-0.02	-0.02	-0.02	0.02	0.04	0.00
Penetration[c] at 25°C	41	47	37	43	40	43

[a] Liner covered with 1.5 in. of silica sand on which the waste was placed.

[b] ASTM E-96, Method BW.

[c] Calculated from viscosity at 0.05 s^{-1} by formula of Carre and Laurent (Association Francaise des Techniciens du Petrole, Bulletin No. 157, pp 1-54, January 31, 1963):

$$(\mathrm{Pen})^{2.6} = \frac{9.5 \times 10^{10}}{\eta}$$

Note: The original design of the project was to expose 12 different liners including soil, admix, sprayed-on, and polymeric membrane liners exposed for two time periods in six different waste sludges.

TABLE 34. POLYMERIC MEMBRANE LINERS SELECTED FOR EXPOSURE IN HAZARDOUS WASTES

Polymer	Thickness, mil
Butyl rubber (IIR), fabric-reinforced	34
Chlorinated polyethylene (CPE)	32
Chlorosulfonated polyethylene (CSPE), fabric-reinforced	34
Elasticized polyolefin (ELPO)	25
Ethylene propylene rubber (EPDM)	50
Neoprene (polychloroprene - CR)	34
Polyester elastomer (PEL)	8
Polyvinyl chloride (PVC)	30

Preliminary Compatibility Testing

A preliminary compatibility test was run on the selected liners plus some additional liners in the available wastes. This test is described in the Interim Report (Haxo et al, 1977). Basically, the test involved the immersion of strips of different lining materials in the different wastes and observing the effects on the strips with time. Major properties that were of importance were the swelling of the membranes in the different wastes and the visual and general effects upon properties as would be measured essentially by hand. Table 35 summarizes the preliminary compatibility tests that were performed prior to selection of membranes to expose as primary liners in the cells. The table also includes data on the asphaltic liner materials and the various sealing materials that were being considered for use in the installation of the liner specimens in the cells. Materials that swelled inordinately or became sticky were eliminated. The final selection of the combination of wastes and polymeric membranes is given in Table 36.

In addition to those combinations of wastes and liners which were tested in two cells, additional single cells were prepared to use for the immersion testing of polymeric materials in additional wastes. These combinations are also shown in Table 36.

Monitoring the Cells

During exposure, the cells were checked for seepage below the liners and for changes in the waste levels in the tanks above the liners.

TABLE 35. EFFECT OF IMMERSION IN WASTE ON MEMBRANE LINERS AND SEALING MATERIALS - PRELIMINARY SCREENING STUDY[a]

Material		Wastes							
		Acidic		Alkaline		Lead		Oily	Pesticide
Composition	Identifiction number[b]	"HFL" (W-10)	"HNO_3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	Low Pb washing (W-8)[c]	Gasoline wash water (W-3)[c]	"Weed oil" (W-7)	"Weed killer" (W-11)
Polymer lining materials									
Butyl rubber (IIR)	22	NVC	NVC	NVC	NVC	NVC	NVC	68	NVC
	24	NVC	NVC	NVC	NVC	NVC	NVC	91	...
	44	...	...	...	...	...	...	76	...
	57R[d]	NVC	NVC	NVC	NVC	NVC	NVC	67	NVC
Chlorinated polyethylene (CPE)	12[d]	NVC	NVC	NVC	NVC	NVC	NVC	RVD	NVC
	38	NVC	NVC	NVC	NVC	NVC	NVC	RVD 92	NVC
	39R	NVC	NVC	NVC	NVC	NVC	NVC	RVD	NVC
	48R	...	...	...	...	...	...	RVD[e]	NVC
	73R	NVC	NVC	NVC	NVC	BLS	NVC	...	...
Chlorosulfonated polyethylene (CSPE)	6R[d]	NVC	NVC	NVC	NVC	NVC	NVC	RVD	NVC
	50RC	...	...	...	...	...	...	6	...
	55	NVC	NVC	NVC	NVC	NVC	NVC	...	...
Elasticized polyolefin (ELPO)	36[d]	NVC	NVC	NVC	NVC	NVC	NVC	49	NVC
	41	NVC	NVC	NVC	NVC	NVC	NVC	42	NVC
Ethylene propylene rubber (EPDM)	8	NVC	NVC	NVC	NVC	NVC	NVC	75	...
	26[d]	NVC	NVC	NVC	NVC	NVC	NVC	90	NVC
Neoprene (CR)	9	CC	SCR	CC	NVC	NVC	NVC	111	...
	37	NVC	NVC	NVC	NVC	NVC	NVC	67	NVC
	42R	NVC	RVD	NVC	NVC	NVC	NVC	REV	NVC
	43R[d]	...	...	...	...	...	...	10	NVC
	47R	...	...	...	...	...	...	6	...
	56R	NVC	NVC	SHD	NVC	NVC	NVC	100	NVC
	74R	NVC	NVC	f	NVC	NVC	NVC	NVC	...
Polyester elastomer (PEL)	69	NVC	NVC	g	NVC	NVC	NVC	12	NVC
	75[d]	NVC	NVC	g	NVC	NVC	NVC	18	NVC
Polyvinyl chloride (PVC)	10	CC	NVC	HDS	NVC	NVC	NVC	NVC	NVC
	11	NVC	NVC	HDS	NVC	NVC	NVC	NVC	NVC
	17	NVC	NVC	HDS	NVC	NVC	NVC	+11	NVC
	40	NVC	NVC	HDS	NVC	NVC	NVC	+24	NVC
	49R	...	...	...	...	...	...	+3	...
	59[d]	NVC	NVC	HDS	NVC	NVC	NVC	+26	NVC
	67	NVC	NVC	HDS	NVC	NVC	NVC	+5	NVC
	71R	NVC	h	HDS	NVC	NVC	NVC	CC	NVC
Polyurethane	45R	...	...	...	...	...	...	50	...
	46R	...	...	...	...	...	...	44	...
	51R	...	...	...	...	...	...	2	...
	70R	CC	NVC	CC	NVC	CC	NVC	33	NVC
	72R	CC	NVC	CC	NVC	NVC	NVC	28	...
Asphaltic membranes									
Coal tar pitch + PVC	52	NVC	NVC	HDS	NVC	NVC	NVC	17	NVC
Emulsified asphalt on nonwoven fabric	58	NVC	SCR, BLS	NVC	NVC	NVC	NVC	RVD	BLS
Sealing materials									
Butyl caulk	63	NVC	NVC	NVC	NVC	NVC	NVC	RVD	...
Neoprene sponge	34	NVC	SCR	SHR	NVC	SHR	SHR	38	SHR
Polyurethane caulk	64	...	RVD	SFT-1&2	NVC	NVC	BLS	SFT-1&2	...
Polysulfide caulk	66	NVC	SFT-1&2	SCR	NVC	NVC	NVC	NVC	...
Teflon sponge rod	68	NVC	NVC	NVC	NVC	NVC	NVC	ABS	...

[a] Based on Table 14 from the Interim Report (Haxo et al, 1977); the unit used is an estimated percent increase in area of that portion of strip immersed.
[b] R = fabric-reinforced; RC = fabric-reinforced and crosslinked.
[c] Blended to make W-14, lead waste.
[d] Materials incorporated in cells as primary liners.
[e] Complete loss of tear strength.
[f] Neoprene in satisfactory condition but reinforcing fabric dissolved.
[g] Black rubs off, indicating possible dissolving of the liner.
[h] 0.10-in. swell in each direction, curled, and delaminated.

KEY TO OBSERVATIONS

ABS - Absorbed waste.
BLS - Blistering of surface.

KEY TO OBSERVATIONS, cont'd

CC - Color change.
HDS - Hardened and shrank.
NVC - No visible change.
REV - Reverted.
RVD - Removed specimen from waste because of excessive swelling or disintegration.
SCR - Surface cracking and hardening.
SFT-1 - Softened above waste.
SFT-2 - Softened in waste.
SHD - Slightly hardened.
SHR - Shrank.

TABLE 36. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS

Polymer	Liner number[b]	Number of cells	Wastes[a]								
			Acidic		Alkaline			Oily			Pest-icide
			"HFL" (W-10)	"HNO$_3$-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	"Lead waste" (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	"Weed killer" (W-11)
Butyl rubber	57	8	-	2	-	2	2	-	-	-	2
Chlorinated polyethylene	77	10	-	2	-	2	2	-	2	-	2
Chlorosulfonated polyethylene	6	10	-	2	-	2	2	-	2	-	2
Elasticized polyolefin	36	15	1	2	1	2	2	2	2	1	2
Ethylene propylene rubber	26	8	-	2	-	2	2	-	-	-	2
Neoprene	43	8	-	-	-	2	2	-	2	-	2
Polyester elastomer	75	12	-	2	-	2	2	2	2	-	2
Polyvinyl chloride	59	15	1	2	1	2	2	2	2	1	2

[a] Matrecon waste serial number shown below identification.

[b] Matrecon liner serial number.

No leakage that could be attributed to seepage through the liners or through seams for periods of up to 56 months occurred in any of the cells. Leakage occurred in a few cells through the flanges, and some cells containing the acidic wastes began to leak through the tank walls due to corrosion. This latter failure required dismantling of the cells and recovery of the test specimens. The level of the waste in the tanks tended to drop due to evaporation, even though the cells were covered with a hardboard cover. Either waste or water was added to replenish the quantity lost due to evaporation. Crystals of the "Spent caustic" waste "crept" over the top edge of the tanks and encrusted part of the outside wall of the cells. Crystals formed above the "waterline" of the "Slop water" waste and did not continue to form up the inner wall as had the "Spent caustic" waste.

Dismantling of Cells and Recovery of Liners

The dismantling and recovery of the liner specimens is an important step in exposure testing of lining materials. At this point, the condition of the exposed lining material becomes visible and the changes that have occurred since exposure began can be observed. Dismantling involved the following major steps:

1. Inspection of the cells immediately before dismantling. This step included the collection, measurement, and analysis of any effluent that may have seeped through the liner and the measurement of the waste depth in the tanks.

2. Observation of the condition of the waste and its removal from the cells. The wastes were removed with as little stirring as possible so that observations could be made about any stratification and the settling out of solid constituents. Also, it allowed observations to be made about the material that was in direct contact with the liner, that is, the buildup of solids and its character. At this point some of the liners were photographed.

3. Cleaning the liner and removing solids that had been deposited on the liner. The liner was then photographed in place.

4. Removal of the tank part of the cell and recovery of the liner. The liner was inspected both on the surface facing the waste and the underneath surface facing the glass cloth and the silica gravel to determine if there were any deposits on the bottom of the liner. The condition of the glass cloth and the silica was also checked.

5. Removal and washing of the silica gravel for subsequent measurement of electrical conductivity and pH of the wash water.

6. Photographing the top and bottom of the liner after removal. The liners were then sealed in polyethylene bags to prevent loss of volatiles from the exposed specimens which usually absorbed water and other volatile constituents in the wastes.

Note: Unless the specimen is tested within a few days, polyethylene bags are inadequate to prevent loss of volatiles.

Figure 17 shows a sequence of photographs of the recovery of the polyvinyl chloride specimen (No. 59) exposed to the pesticide "Weed killer" waste for 500 days. The top photograph shows the liner after removal of the waste. The deposit on the liner surface is a clay material that was used as a carrier for the herbicide. The middle photograph shows the liner after it was cleaned and removed from the cell and shows the imprint of the gravel beneath the liner. This liner had a volatiles content of 2.3%, probably mostly water. The photograph also shows the seam which was double layered. The bottom photograph shows the condition of the silica gravel and the glass cloth. In this cell, and as occurred with most of the cells with polymeric membrane liners, the condition of the fabric and the gravel appeared to be essentially the same as they were at the time the exposure was started.

Figure 18 shows two photographs of a neoprene liner (No. 43) that had been exposed to the lead waste for 499 days; it had swelled considerably during the exposure period. The top photograph shows the liner in place in the cell after it had been cleaned. The second photo shows the exposed specimen after it had been removed from the cell. The wrinkling and distortion that are shown took place after the waste had been removed from the specimen, at which time the flat-appearing, exposed specimen billowed and became distorted. This liner had absorbed considerable waste by the time it was dismantled as indicated by its high volatiles content and its flabby appearance and feel.

Generally, the dismantling was performed in groups of cells having the same waste so that the comparisons of the effects of the waste on several lining materials could be made visually and the handling of the disposal of the waste would be more convenient. The times of dismantling for each cell, that is the days after the respective wastes were placed in the cells and thus the days of exposure, are presented in Table 37.

Testing of the Exposed Polymeric Membrane Liners

After removal from the cells and cleaning, the exposed membrane liners were visually inspected thoroughly with low-power magnification for any unusual effects of the waste, such as blistering, delamination, surface deterioration, etc, and then tested as soon as possible. The typical pattern used for dieing out physical test specimens from the exposed liner specimen is shown in Figure 19. This type of pattern was initially used for testing all of the exposed primary liner specimens including the fabric-reinforced sheeting as well as the sheeting without the fabric reinforcement. The special dumbbell with the 0.25-in. wide cross section was used to test the fabric-reinforced sheeting even though it was recognized that this type of test specimen is not appropriate for testing fabric-reinforced sheeting. This dumbbell is described in more detail in the subsection of Section 7 regarding measurements of immersed specimens./ The tear resistance of the fabric-reinforced sheeting was not determined, because the short threads pulled out of the specimens.

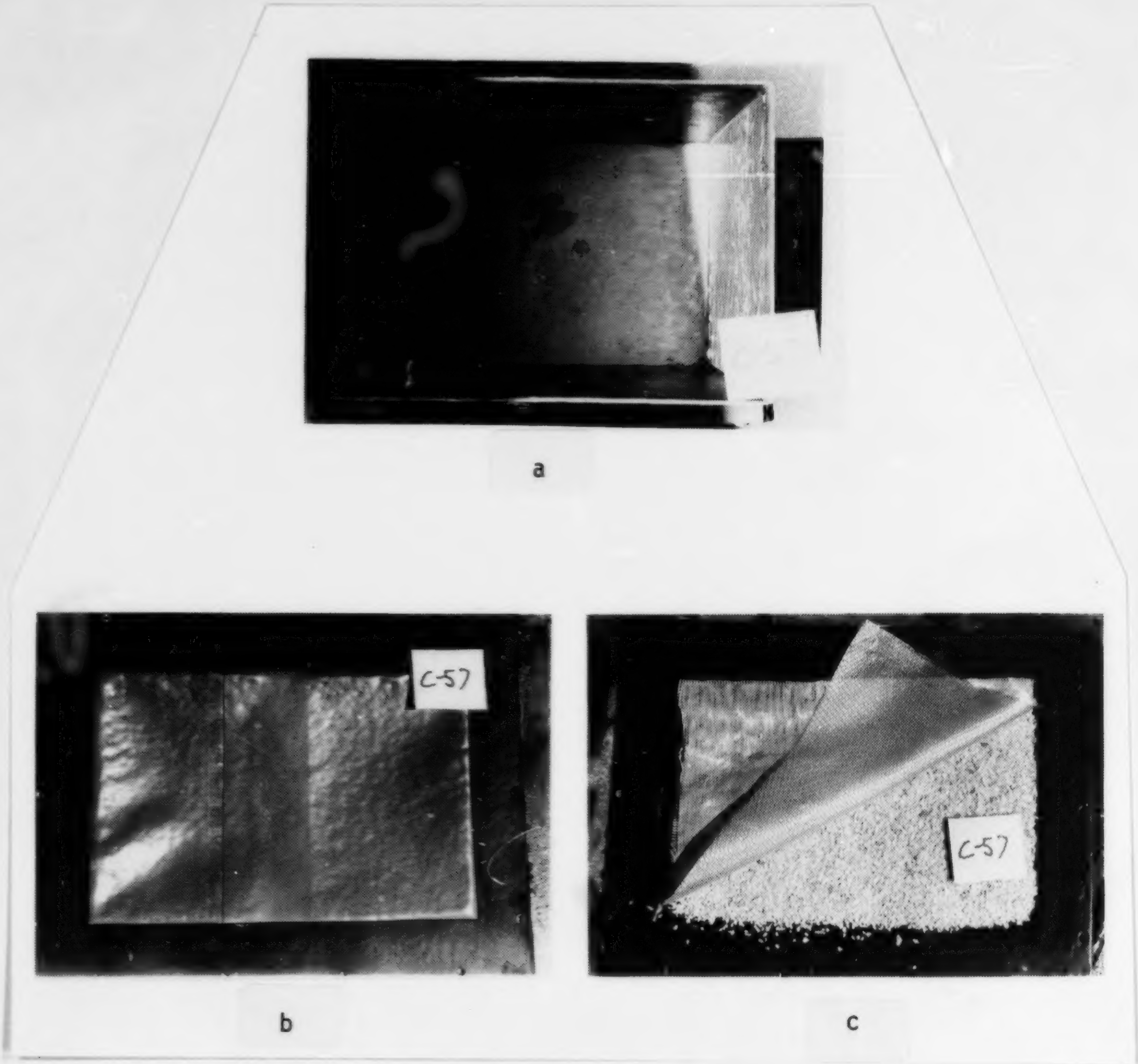


Figure 17. A sequence of photographs showing the recovery of the polyvinyl chloride specimen (No. 59) exposed to the pesticide "Weed killer" waste for 500 days. Fig. 17a shows the liner after the the waste has been removed. The light colored deposit on the bottom is a clay that was used as a carrier for the herbicide. Fig. 17b shows the liner after it was cleaned and removed from the cell with the imprint of the gravel beneath the liner. The photograph also shows the seam which was double layered. Fig. 17c shows the condition of the gravel and the glass cloth.

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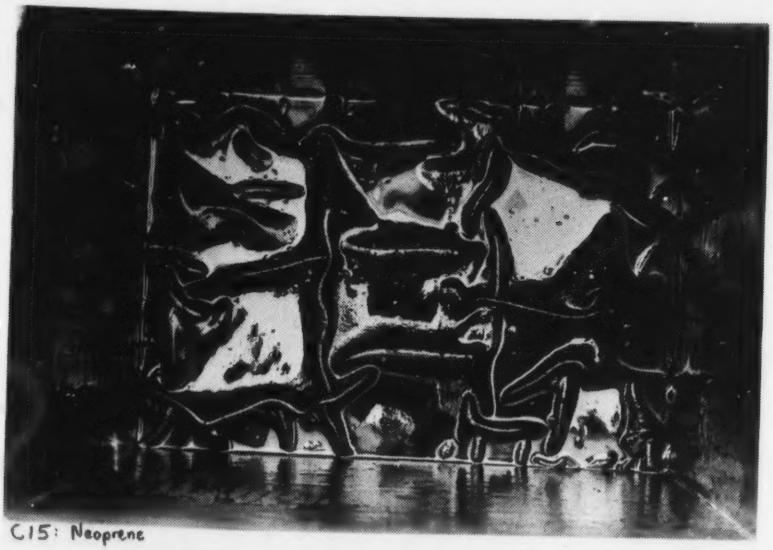
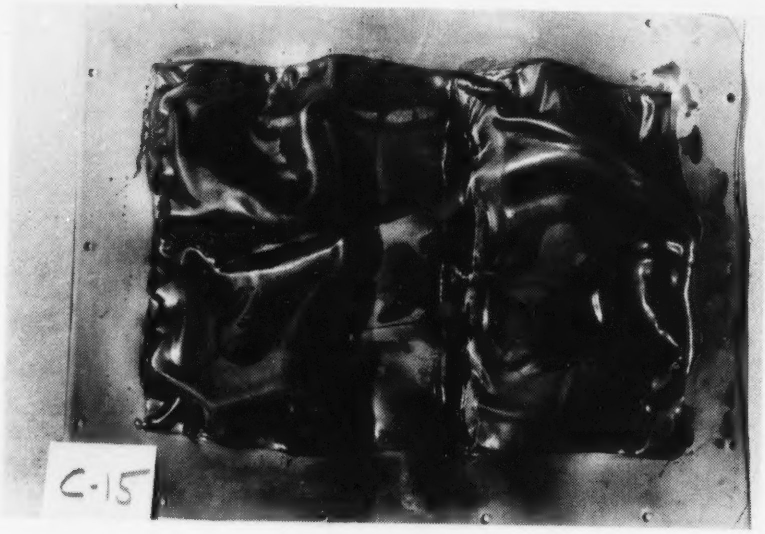


a



b

Figure 18. Two photographs of the recovered neoprene liner (No. 43) that had been exposed to the lead waste for 499 days. Fig. 18a shows the liner in place after it had been cleaned. Fig. 18b shows the liner exposure specimen after removal from the cell.

TABLE 37. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS - DAYS OF EXPOSURE

Polymeric membrane liner		Wastes[a]								
		Acidic		Alkaline			Oily			Pesticide
Polymer	Number[b]	"HFL" (W-10)	"HNO_3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	"Lead waste" (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	"Weed killer" (W-11)
Butyl rubber	57R	... ...	505 1218	... ...	526 1249	499 1339	... ...	... ...	... ...	500 1258
Chlorinated polyethylene	77	... ...	459 1218	... ...	526 1249	499 1334	... ...	521 1358	... ...	500 1258
Chlorosulfonated polyethylene	6R	... ...	505 1218	... ...	526 1249	499 1343	... ...	521 1357	... ...	504 1258
Elasticized polyolefin	36	2293 ...	505 1217	2300 ...	526 2677	499 1343	327 2355	521 1357	... ...	494 2699
Ethylene propylene rubber	26	... ...	497 1147	... ...	526 124	499 1344	... ...	... ...	... ...	500 1258
Neoprene	43	... ...	... ...	... ...	526 1237	499 1342	... ...	521 1356	... ...	494 1257
Polyester elastomer	75	... ...	323 509	... ...	526 1237	499 1342	328 ...	521 1357	... ...	501 1258
Polyvinyl chloride	59	1565 ...	505 1352	1565 ...	526 1249	499 1345	327 ...	521 1356	... ...	500 1258

[a] Matrecon waste serial number shown below identification.

[b] Matrecon liner serial number; R = fabric-reinforced.

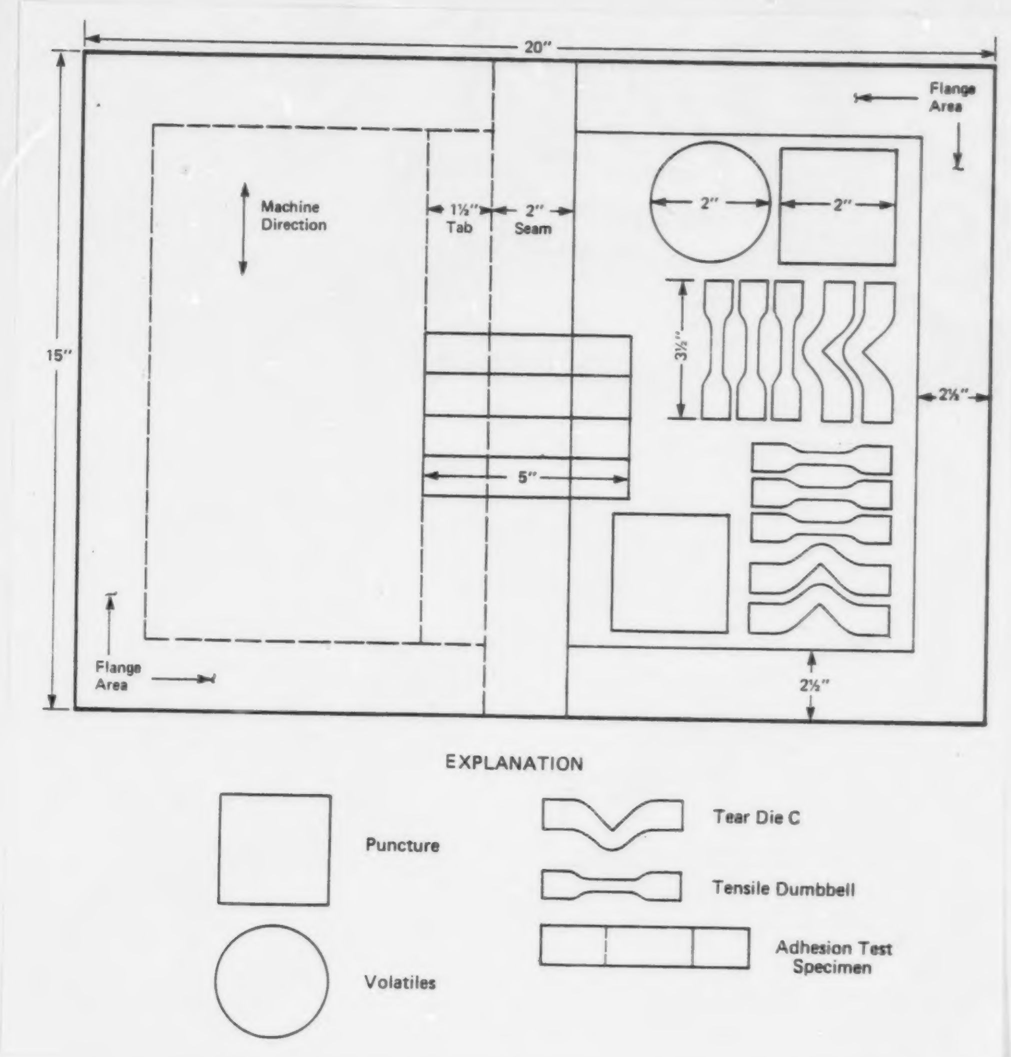

20"
Flange Area
Machine Direction
1½" Tab
2" Seam
2"
2"
15"
3½"
2½"
5"
Flange Area
2½"
EXPLANATION
Puncture
Volatiles
Tear Die C
Tensile Dumbbell
Adhesion Test Specimen

Figure 19. Principal pattern used for dieing out the physical test specimens from the exposed primary exposure specimens.

Most of the fabric-reinforced sheeting made for use in the lining of pits and ponds generally has very low adhesion between the fabric threads and the polymer coating. In other words, the fabric and the coating can act independently. The fabric can break at low elongation without causing the polymer coating to break.

As the primary objective in this project was to determine the effects of exposures to different wastes on the polymer compound and not on the fabric, we were concerned with the coating. The effect on the fabric is a separate problem which involves not only the type of fiber but its size and the weave of the fabric. The use of the two test specimens appeared to be satisfactory for membranes with less than 10 x 10 epi, such as the CSPE sheeting (No. 6R) with 8 x 8 epi nylon fabric. With this construction of membrane, in the tensile test the fabric broke at elongations usually less than 25%. The CSPE coating compound remained as a continuous sheet, which ultimately broke at a much higher elongation, e.g. >200 percent. In the tear test the threads pulled out leaving the coating to tear independently. It was possible to follow the changes in the coating compound during the exposure by considering the breaking and tearing of the coating and neglecting the behavior of the fabric.

However, the more tightly woven fabric (22 x 11 epi) with its slight adhesion to the butyl compound in membrane 57R did not allow the rubber coating to act separately from the fabric. With this membrane (57R), when the fabric broke in the tensile test in the transverse direction, the coating broke almost simultaneously, so the effect of exposure on the coating could not be assessed. The effect upon the tear values was somewhat similar. As a consequence, tear testing of fabric-reinforced membranes was dropped from the test and 1-in. strips of fabric-reinforced membranes were used later in the project for assessing the effect of exposure on tensile properties. In such cases, a different pattern was used to die out the test specimens as shown in Figure 20.

The principal properties that were measured on the exposed membranes were:

- Volatiles content.
- Extractables content.
- Hardness.
- Tensile properties in machine and transverse directions, i.e. tensile strength, elongation at break, and stress at 100% and 200% elongation.
- Tear strength of unreinforced sheeting measured in machine and transverse directions.
- Puncture resistance.
- Seam strength in shear and peel modes.

The results of testing the exposed membranes are presented in Appendix I. These data are presented by individual lining material, by the type of waste, and for each of the two elapsed times that the liners were exposed. Data are presented on the properties listed in Table 15, with the exception of the data on seam strength which are presented in Table 43. Tensile and tear properties which are measured in both machine and transverse

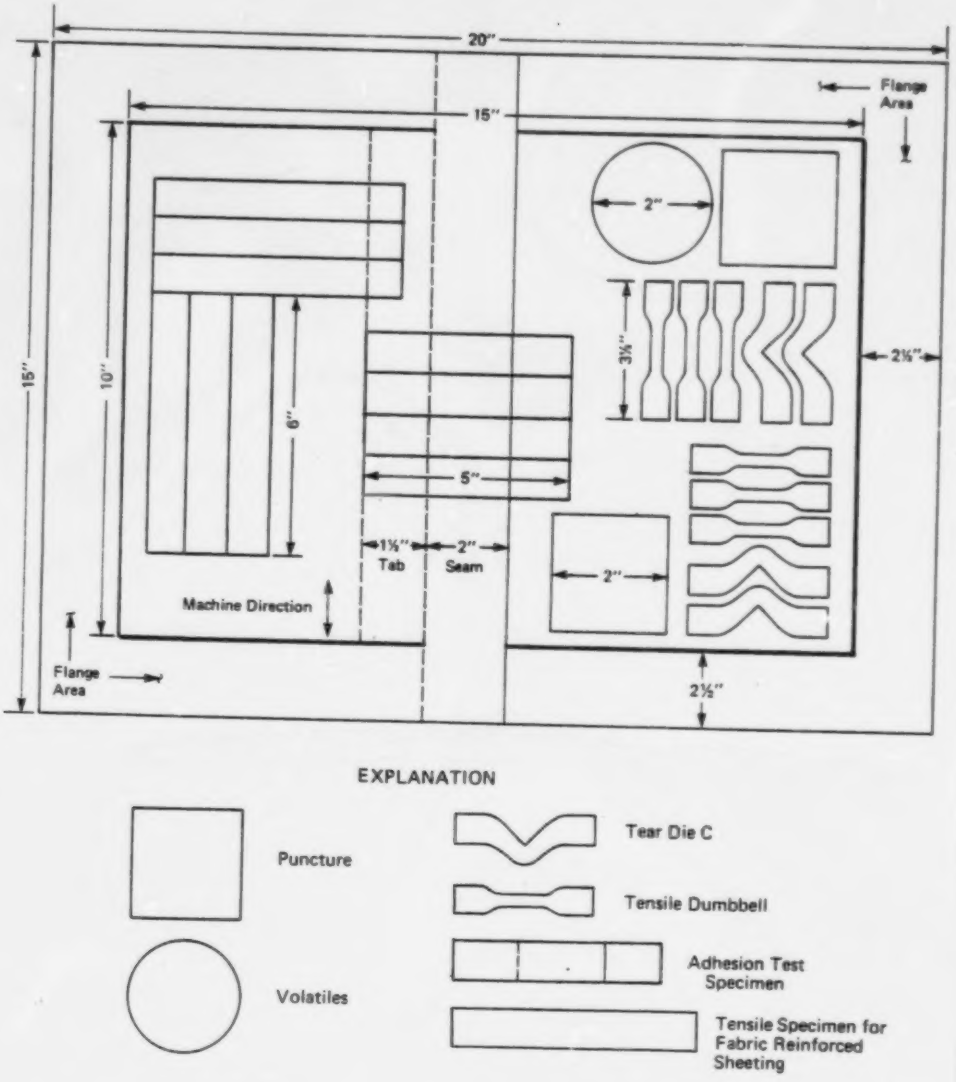

20"
Flange Area
15"
2"
3½"
2½"
15"
10"
6"
5"
1½" Tab
2" Seam
2"
Machine Direction
Flange Area
2½"
EXPLANATION
Puncture
Volatiles
Tear Die C
Tensile Dumbbell
Adhesion Test Specimen
Tensile Specimen for Fabric Reinforced Sheeting

Figure 20. Pattern used for dieing out the physical test specimens from the exposed primary specimens with fabric reinforcement. Both dumbbells and 1-in. strip test specimens were cut and tested in the second set of exposed specimens.

directions were averaged and the averages were used to develop the retention data reported; thus, the test results for all the exposed liners can be compared.

Retention of Selected Properties of Liners on Exposure to Different Wastes

Data on four properties of significance in measuring the effects on polymeric membrane liners of exposure to wastes are presented in Tables 38, 39, 40, and 41 for the exposed materials for each of the respective exposure periods shown in Table 37. These properties are percent volatiles, percent extractables, percent retention of stress at 100% elongation, and percent retention elongation at break. The data show the effect of the waste at each exposure time on each of these properties. Comparisons of the effects of the different wastes on the lining materials can thus be made. The data also show both the variation in magnitude of the effects on different polymeric lining materials by given wastes and the different effects of the different wastes on a given lining material.

Information on the seams and on the retention of seam strength are given in Tables 42 through 44. Table 42 presents information on the type of seaming procedures along with information on the fabricator of the seams. Table 43 presents the results of testing the seam strength of the specimens measured in shear after their respective exposures, together with data on the unexposed materials. Table 44 presents results of testing the strength of the seams in the peel mode. All the data show the strength values in pounds-per-inch width (ppi) and the locus of failure of the adhesion test specimen.

The results of the exposure testing of the individual liners are discussed in subsequent subsections.

Butyl Rubber Membrane--

The butyl rubber membrane (No. 57R) was fabric reinforced with a nylon scrim of 22 x 11 epi thread count. It had a nominal thickness of 34 mils with a vulcanized coating compound that contained 23.46% ash, 6.36% extractables, and 0.29% volatiles. The high ash content reflects the use of inorganic fillers in the compound. Two sets of specimens of this membrane were exposed to four wastes:

Acidic waste, "HNO_3-HF-HOAc"
Alkaline waste, "Spent caustic"
Lead waste
Pesticide waste.

Combinations of the butyl membrane with the oily wastes were not selected for the primary exposure program as a result of the preliminary compatibility testing. The lead waste was included because, given the results of the compatibility testing, it appeared to contain only a small amount of oily constituents.

The specimens from the first set were recovered after 500 to 526 days of exposure and specimens from the second set after 1218 to 1339 days of exposure.

TABLE 38. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS - PERCENT VOLATILES[a]

Polymeric membrane liner		Original value, %	Wastes[b]								
			Acidic		Alkaline			Oily			Pesticide
Polymer	Number[c]		"HFL" (W-10)	"HNO3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	"Lead waste" (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	"Weed killer" (W-11)
Butyl rubber	57R	0.29	... ...	5.92 11.46	... ...	1.75 1.37	2.79 3.53	... ...	... ...	... ...	4.10 4.79
Chlorinated polyethylene	77	0.14	... ...	7.82 13.18	... ...	2.32 2.79	11.58 19.20	... ...	3.69 10.11	... ...	4.99 7.91
Chlorosulfonated polyethylene	6R	0.51	... ...	4.69 7.18	... ...	4.77 5.77	1.08 11.44	... ...	7.51 10.25	... ...	8.00 9.73
Elasticized polyolefin	36	0.15	1.46 ...	3.20 5.26	10.83 ...	1.25 1.01	1.03 1.53	0.38 4.02	2.15 5.12	... ...	0.13 0.58
Ethylene propylene rubber	26	0.50	... ...	8.95 12.02	... ...	1.27 1.31	2.83 5.25	... ...	... ...	... ...	3.34 6.29
Neoprene	43	0.45	... ...	... ...	... ...	4.40 5.67	18.01 17.50	... ...	12.99 21.31	... ...	11.29 13.63
Polyester elastomer	75	0.26	... ...	0.39 4.74	... ...	0.65 0.89	2.63 1.72	0.40 ...	1.27 2.59	... ...	0.60 2.92
Polyvinyl chloride	59	0.31	9.90 ...	12.08 13.94	18.72 ...	2.34 1.85	3.34 4.43	0.29 ...	1.70 4.19	... ...	2.30 3.61

[a] Respective durations of exposure are presented in Table 37.

[b] Matrecon waste serial number shown below identification.

[c] Matrecon liner serial number; R = fabric reinforced.

TABLE 39. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS - PERCENT EXTRACTABLES[a]

Polymeric membrane liner		Original value,	Wastes[b]								
			Acidic		Alkaline			Oily			Pesticide
			"HFL"	"HNO$_3$-HF-HOAc"	"Slop water"	"Spent caustic"	"Lead waste"	"Slurry oil"	"Oil Pond 104"	"Weed oil"	"Weed killer"
Polymer	Number[c]	%	(W-10)	(W-9)	(W-4)	(W-2)	(W-14)	(W-15)	(W-5)	(W-7)	(W-11)
Butyl rubber	57R	6.36	...	...	...	...	7.75	...	...	...	5.15
			...	8.65	...	7.86	7.86	...	...	...	7.62
Chlorinated polyethylene	77	9.13	...	10.09	...	...	7.31	...	...	...	9.72
			...	9.41	...	9.10	7.24	...	17.00	...	9.41
Chlorosulfonated polyethylene	6R	3.77	...	...	...	4.77	3.52	...	...	...	4.13
			...	4.62	...	5.77	5.95	...	9.45	...	5.39
Elasticized polyolefin	36	5.50	5.40	5.40	1.70	...	5.66	13.94	13.72	...	7.14
			...	7.09	...	5.96	8.06	23.88	20.74	...	6.86
Ethylene propylene rubber	26	22.96	...	21.36	...	...	22.27	...	...	...	23.13
			...	...	...	23.95	26.01	...	...	...	25.20
Neoprene	43	13.69	...	...	...	...	12.54	...	...	...	13.25
			...	...	...	13.69	12.15	...	15.86	...	16.14
Polyester elastomer	75	2.74	...	10.77	...	3.85	2.98	9.91	5.68	...	5.15
			...	13.36	...	3.31	5.35	...	7.28	...	5.83
Polyvinyl chloride	59	35.86	34.42	16.68	10.40	34.62	33.47	39.63	32.62	...	35.27
			...	18.58	...	35.61	22.47	...	29.99	...	33.39

[a] Respective durations of exposure are presented in Table 37.

[b] Matrecon waste serial number shown below identification.

[c] Matrecon liner serial number; R = fabric-reinforced.

TABLE 40. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS - PERCENT RETENTION OF ELONGATION AT BREAK[a]

Polymeric membrane liner		Original value, %	Wastes[b]								
			Acidic		Alkaline			Oily			Pesticide
Polymer	Number[c]		"HFL" (W-10)	"HNO_3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	"Lead waste" (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	"Weed killer" (W-11)
Butyl rubber	57R	42[d]	... ...	60 645	... ...	60 219	119 167	... ...	... ...	... ...	143 100
Chlorinated polyethylene	77	402	... ...	89 89	... ...	107 88	101 83	... ...	98 88	... ...	100 89
Chlorosulfonated polyethylene	6R	242	... ...	90 79	... ...	70 65	107 77	... ...	103 72	... ...	112 85
Elasticized polyolefin	36	665	98 ...	99 96	88 ...	100 97	92 94	96 97	86 78	... ...	101 97
Ethylene propylene rubber	26	450	... ...	97 94	... ...	102 95	100 106	... ...	... ...	... ...	100 104
Neoprene	43	320	... ...	... ...	... ...	98 95	76 75	... ...	86 92	... ...	93 83
Polyester elastomer	75	575	... ...	<1 4	... ...	86 86	98 90	77 ...	95 92	... ...	96 87
Polyvinyl chloride	59	375	88 ...	82 71	3 ...	102 97	95 93	90 ...	85 85	... ...	101 95

[a] Respective durations of exposure are presented in Table 37.

[b] Matrecon waste serial number shown below identification.

[c] Matrecon liner serial number; R = fabric-reinforced.

[d] Value reported is elongation at ultimate break with respective retention values. Fabric in sheeting retained its original elongation (25%).

TABLE 41. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS - PERCENT RETENTION OF STRESS AT 100% ELONGATION[a]

Polymeric membrane liner		Original value, psi	Wastes[b]								
			Acidic		Alkaline			Oily			Pesticide
Polymer	Number[c]		"HFL" (W-10)	"HNO$_3$-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	"Lead waste" (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	"Weed killer" (W-11)
Butyl rubber	57R	(d)	...	...	...	...	...	...	...	...	...
Chlorinated polyethylene	77	900	... ...	97 113	... ...	82 129	56 71	... ...	53 62	... ...	94 113
Chlorosulfonated polyethylene	6R	937	... ...	106 112	... ...	165 200	85 118	... ...	63 96	... ...	90 118
Elasticized polyolefin	36	922	98 ...	92 98	97 ...	87 87	95 104	70 60	61 71	... ...	104 91
Ethylene propylene rubber	26	357	... ...	81 70	... ...	88 108	84 80	... ...	... ...	... ...	89 87
Neoprene	43	460	... ...	... ...	... ...	90 95	53 61	... ...	50 42	... ...	62 54
Polyester elastomer	75	2585	... ...	... ...	... ...	101 109	88 88	77 ...	94 85	... ...	95 96
Polyvinyl chloride	59	995	153 ...	200 249	... ...	99 115	82 103	113 ...	152 174	... ...	100 137

[a] Respective durations of exposure are presented in Table 37.

[b] Matrecon waste serial number shown below identification.

[c] Matrecon liner serial number; R = fabric-reinforced.

[d] Unexposed material broke at less than 100% elongation.

TABLE 42. SEAMS IN POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS

Polymeric membranes	Liner number	Method of seaming	Seam width, in.	Fabricator
Butyl rubber	57R	Vulcanizable adhesive furnished by supplier of liner	2	Matrecon
Chlorinated polyethylene	77	Solvent weld with mixture of 1 part toluene and 1 part tetrahydrofuran	2	Matrecon
Chlorosulfonated polyethylene	6R	Adhesive furnished by liner supplier	2	Matrecon
Elasticized polyolefin	36	Heat sealed	0.5	Supplier
Ethylene propylene rubber	26	Adhesive and gum tape furnished by supplier	2	Matrecon
Neoprene	43	Cement and lap sealant furnished by supplier of liner	2	Matrecon
Polyester elastomer	75	Heat sealed	0.5	Supplier
Polyvinyl chloride	59	Solvent weld using mixture of 2 parts tetrahydrofuran and 1 part cyclohexanone	2	Matrecon

[a] All seams were allowed to age at least a month before being tested or covered with wastes.

TABLE 43. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS - EFFECT ON SEAM STRENGTH MEASURED IN SHEAR MODE[a]

Polymeric membrane liner		Original value,	Method of	Seam strength in ppi after exposure to different wastes[b]								
				Acidic		Alkaline			Oily			Pesticide
				"HFL"	"HNO_3-HF-HOAc"	"Slop water"	"Spent caustic"	"Lead waste"	"Slurry oil"	"Oil Pond 104"	"Weed oil"	"Weed killer"
Polymer	Number[c]	ppi	seaming	(W-10)	(W-9)	(W-4)	(W-2)	(W-14)	(W-15)	(W-5)	(W-7)	(W-11)
Butyl rubber	57R	>84.8[d]	Cement[e]	... ...	>67.8[d] >73.3[d]	... ...	>74.4[f] >78.6[d]	64.6[g] >70.7[d]	... ...	... ...	... ...	69.1[h] >68.8[d]
Chlorinated polyethylene	77	48.3[g]	Solvent	... ...	27.1[h] 39.8[h]	... ...	41.5[h] 46.0[h]	26.3[i] 26.3[g]	... ...	16.4[g] 25.5[g]	... ...	>38.8[d] >44.5[d]
Chlorosulfonated polyethylene	6R	62.1[g]	Cement	... ...	52.7[g] 57.4[g]	... ...	64.0[i] >76.3[d]	58.2[j] 66.6[g]	... ...	60.5[h] 60.3[d]	... ...	>61.3[d] 65.7[h]
Elasticized polyolefin	36	28.8[g]	Heat[k]	32.4[g] ...	29.2[g] 32.0[g]	31.3[h] ...	28.9[g] 31.4[g]	26.0[g] 29.0[g]	21.7[d] 24.4[g]	>19.3[g] 18.3[g]	... ...	26.5[g] 32.5[g]
Ethylene propylene rubber	26	39.0[l]	Cement[e]	...	47.9[l] ...	... ...	39.1[l] 45.9[m]	32.2[l] 13.9[l]	... ...	... ...	... ...	46.5[l] 44.4[l]
Neoprene	43	45.7[n]	Cement[e]	... ...	... ...	... ...	>51.9[o] >58.5[d]	>31.6[o] 27.1[n]	... ...	24.8[p] 14.6[g]	... ...	>23.7[d] 34.9[h]
Polyester elastomer	75	21.2[g]	Heat[k]	... ...	... ...	... ...	... ...	25.2[h] >16.7[d]	>18.8[d] ...	28.2[g] >14.6[d]	... ...	26.5[g] 21.9[h]
Polyvinyl chloride	59	>69.1[d]	Solvent	69.2[g] ...	74.7[g] 79.0[h]	71.6[q] ...	50.4[g] 63.6[g]	53.1[g] 45.0[g]	59.3[g] ...	60.9[g] 77.1[g]	... ...	46.0[i] 60.4[g]

[a] Strip specimen 1 in. wide; initial jaw separation, 4 in.; rate of jaw separation, 2 ipm. All seams fabricated by Matrecon following manufacturers' recommendations, except where otherwise noted. Value for seam strength is reported in pounds-per-inch-width (ppi). A "greater than" symbol is used to indicate that the strength of the seam itself is greater than the value reported. See Table 37 for durations of exposure.

[b] Matrecon waste serial number shown below identification.

[c] Matercon liner serial number; R = fabric-reinforced.

[d] Specimens failed at clamp edge.

[e] Low-temperature vulcanizing adhesive.

[f] Specimens failed outside of seam area and not in the clamped area.

[g] Specimens failed at seam edge.

[h] One specimen failed at seam edge; the other specimen failed in the clamped area.

[i] One specimen failed at seam edge; the other failed outside of seam area and not in the clamped area.

[j] One specimen failed outside seam area; the other failed in seam area which had been separated.

[k] Seam fabricated by supplier.

[l] Specimens failed in adhesive.

[m] One specimen failed at seam edge; other failed in adhesive.

[n] Specimens failed in bond between adhesive and liner surface.

[o] One specimen failed at clamp edge; other failed outside of seam area and not in clamped area.

[p] One specimen failed at clamp edge; other failed in bond between adhesive and liner surface.

[q] Two specimens failed outside seam area; one specimen failed at clamp edge; two failed at seam edge.

TABLE 44. EXPOSURE OF POLYMERIC MEMBRANE LINER SPECIMENS IN PRIMARY CELLS - EFFECT ON SEAM STRENGTH MEASURED IN PEEL MODE[a]

Polymeric membrane liner		Original value, ppi	Method of seaming	Seam strength in ppi after exposure to different wastes[b]								
				Acidic		Alkaline			Oily			Pesticide
Polymer	Number[c]			"HFL" (W-10)	"HNO$_3$-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	"Lead waste" (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	"Weed killer" (W-11)
Butyl rubber	57R	8.0[d]	Cement[e]	... ...	3.2[f] 4.5[f]	... ...	8.5[f] 4.1[f]	7.8[f] 5.3[f]	... ...	... ...	... ...	10.9[f] 3.6[f]
Chlorinated polyethylene	77	21.4[g]	Solvent	... ...	17.9[g] 19.0[g]	... ...	21.9[g] 21.0[h]	17.3[g] 13.7[g]	... ...	14.3[i] 11.4[g]	... ...	20.0[g] 16.2[g]
Chlorosulfonated polyethylene	6R	23.2[j]	Cement	... ...	14.1[f] 12.5[k]	... ...	21.3[f] 28.4[f]	26.4[j] 21.5[k]	... ...	16.5[f] 15.0[f]	... ...	21.3[f] 15.1[f]
Elasticized polyolefin	36	21.0[l]	Heat[m]	22.6[l] ...	21.5[l] 22.0[l]	22.0[l] ...	19.8[l] 22.0[l]	19.8[l] 22.2[l]	18.7[l] 18.6[l]	>14.5[l] 14.9[l]	... ...	20.9[l] 23.2[l]
Ethylene propylene rubber	26	4.9[k]	Cement[e]	... ...	5.9[k] ...	... ...	4.5[k] 5.4[k]	3.5[k] 2.3[k]	... ...	... ...	... ...	5.0[k] 5.0[k]
Neoprene	43	8.2[n]	Cement[e]	... ...	... ...	... ...	8.8[n] 10.8[n]	3.3[n] 2.2[n]	... ...	2.1[n] 3.6[n]	... ...	7.6[n] 4.3[n]
Polyester elastomer	75	20.8[g]	Heat[m]	... ...	... ...	... ...	... ...	17.4[o] 18.3[p]	18.2[l] ...	18.8[l] 16.2[q]	... ...	20.3[l] 19.5[l]
Polyvinyl chloride	59	15.2[g]	Solvent	23.9[r] ...	28.4[s] 34.0[g]	9.8[r] ...	21.4[g] 14.8[g]	19.0[s] 16.4[r]	20.3[g] ...	23.1[s] 22.0[r]	... ...	18.1[g] 23.0[r]

[a] Strip specimen 1-in. wide; initial jaw separation, 2 in.; rate of jaw separation, 2 ipm. Value reported in pounds-per-inch-width (ppi) is average after initial peak, except where otherwise noted. All seams fabricated by Matrecon following manufacturers' recommendations, except where otherwise noted. See Table 37 for durations of exposure.

[b] Matrecon waste serial number shown below identification.

[c] Matercon liner serial number; R = fabric-reinforced.

[d] Specimens failed in adhesive.

[e] Low-temperature vulcanizing adhesive.

[f] Specimens failed by a combination of failure in the adhesive and in delamination of the lining material.

[g] Specimens failed at bond between the two liner sufaces.

[h] Specimens initially failed at bond between the two liner surfaces, then failed catastrophically at the line of peel in the course of the test.

[i] One specimen failed catastrophically at the line of peel shortly after it began to peel; the other failed at bond between the two liner surfaces.

[j] One specimen failed by delamination of lining material; the other by failure in the adhesive.

[k] Specimens failed in the adhesive.

[l] Specimens failed catastrophically across the width of the specimen at the line of peel after peeling approximately 0.1 in. Values reported are maximum stresses immediately before catastrophic failures.

[m] Seam fabricated by supplier.

[n] Specimens failed in bond between adhesive and liner surface.

[o] Specimens ripped uncontrollably once peel was initiated. Value reported is maximum stress.

[p] One specimen failed at jaw bite; the other failed catastophically at the line of peel after peeling approximately 0.1 in.

[q] Specimens failed at jaw bite.

[r] Specimens initially failed at the bond between the two liner surfaces, then failed by ripping uncontrollably.

[s] One specimen failed at the bond between the two liner surfaces; the other initially failed at the bond between the two surfaces, then ripped uncontrollably.

Because of the high thread count, 22 x 11 epi, the unexposed butyl rubber sheeting broke with the fabric at 60% elongation in the machine direction and at 25% in the transverse direction; thus retention values could not be calculated for stress at 100% elongation (S-100).

This sheeting had a volatiles content (predominantly moisture) that was particularly high after exposure to the acidic waste and modest after exposure to the pesticide waste, probably reflecting the high inorganics content of the butyl compound (Table 38). The extractables as shown in Table 39 did not change appreciably, indicating that the compounding oil was retained during the exposure to the wastes. Exposure to an oily waste would probably have resulted in a significant increase in extractables. The sheeting appeared to have softened considerably in the acidic waste and developed a high elongation as shown in Table 40. In the other wastes the elongation increased. The softening of the butyl did not appear to affect the nylon scrim.

The 2-in. seam of the butyl rubber specimen was made with a two-part vulcanizable adhesive furnished by the supplier. The unexposed seam failed in the shear mode in the clamp indicating the seam strength was greater than 84.8 ppi. The exposed samples failed predominantly at the clamps also and test values were somewhat less, possibly indicating a loss in the strength of the nylon fabric.

In the peel mode, the original adhesion was 8 ppi with the failure in the adhesive, but the exposed samples delaminated at values of 3.6 to 5.3 ppi. This type of failure indicates loss in ply adhesion during exposure to the wastes.

Overall, except for peel adhesion, the butyl rubber specimens showed good retention of their original properties on exposure to the four wastes. The effect of time was not large. The waste which caused the greatest change, perhaps, was the acidic waste in which the butyl increased in volatiles content significantly. It should be recognized that this liner was not exposed to an obviously oily waste, which would have caused softening and loss of tensile.

Chlorinated Polyethylene (CPE) Membrane--

The CPE (No.77) was a thermoplastic sheeting of 30 mil nominal thickness and was not fabric-reinforced. It had an original ash content of 12.56% and an extractables content of 9.13%. The volatiles of the unexposed material was 0.14%. Two sets of specimens of this sheeting were exposed to five wastes:

Acidic waste, "HNO_3-HF-HOAc"
Alkaline waste, "Spent caustic"
Lead waste
Oily waste, "Oil Pond 104"
Pesticide waste.

The first set of specimens was removed from exposure after 450 to 526 days and the second set after 1218 to 1358 days.

The CPE membrane showed significant increases in volatiles content in the acidic, lead, and pesticide wastes, probably reflecting the absorption of water. The increase during exposure to "Oil Pond 104" waste was probably due to absorption of oil as well as of water. The smallest increase in volatiles was in the specimen exposed to the spent caustic. The modulus in all cases showed an initial drop and then an increase, indicating initial swelling followed by crosslinking. However, there were losses in modulus in the lead waste and in the oily waste. The only significant increase in the extractables during exposure was in the CPE liner exposed to the oily waste, "Oil Pond 104."

The 2-in. seams of the CPE specimens were prepared by Matrecon using a solvent weld that was a 1:1 mixture of toluene and tetrahydrofuran (THF). The unexposed seam had a strength in shear of 48.3 ppi, and failed at the seam edge. The exposed samples generally had lower adhesion, reflecting the loss in strength of the CPE liner during exposure. The failures were at both the seam edge and in the clamp area. Again, the oily wastes appear to reduce the values, which is a reflection of the loss in the strength of the sheeting.

The unexposed seam had a strength in the peel mode of 21.4 ppi with failure at the bond between the two liner surfaces. On exposure, most of the failures were between the two liner surfaces, although one of the exposed specimens in the "Oil Pond 104" waste and one in the "Spent caustic" waste failed catastrophically at the peel line during the test. It appears that the oily waste affected somewhat the strength of the seam.

Overall, in this exposure test, the CPE membrane appeared to be satisfactory for the inorganic aqueous solutions but showed significant losses in properties in contact with oily wastes.

Chlorosulfonated Polyethylene (CSPE) Membrane--

The CSPE membrane (No. 6R) was a fabric-reinforced nylon with a thread count of 8 x 8 epi and a thickness of 34 mils. The CSPE compound contained 3.28% ash, had an extractables content of 3.77%, and a volatiles content of 0.51%. Two sets of specimens of this polymeric membrane were exposed to five wastes:

Acidic waste, "HNO_3-HF-HOAc"
Alkaline waste, "Spent caustic"
Lead waste
Oily waste, "Oil Pond 104"
Pesticide waste.

The first set of specimens was removed after 499 to 526 days of exposure and the second set after 1218 to 1357 days of exposure.

On exposure all the CSPE membrane specimens increased significantly in volatile contents (Table 38) in all the wastes. Though volatiles of the CSPE liner increased in the spent caustic and pesticide wastes, the magnitude appeared to be leveling off at the time the second set was removed. The extractable (Table 39) changed only modestly during the exposure. The highest extractable content measured after exposure was for the liner that had been exposed to "Oil Pond 104" waste; in this case, the extractables increased from 3.77 to 9.45%. The elongation at break of the sheeting decreased with exposure time in all cases, indicating that crosslinking was taking place. This is confirmed by the increases in the S-100.

The seam of the CSPE membrane was prepared by Matrecon using a bodied solvent adhesive furnished by the supplier. The unexposed 2-in. seam had a strength in shear of 62.1 ppi with failure at the seam edge. After exposure, the shear values were generally somehwat lower with the locus of failures varying from the seam edge to the edge of the clamps with some failing in the sheeting itself. The greatest drop in seam strength values was with the seams exposed to the acidic waste; these drops probably reflect the loss in strength of the nylon fabric.

The strength of the unexposed seam in the peel mode was 23.2 ppi; one specimen failed by delamination and the other failed in the adhesive. After exposure, there was little or no loss in the strength of the membranes exposed in the "Spent caustic" and the lead waste although failures were partially by delamination; the principal loss in strength of the test specimens were in those exposed to the acidic waste, "Oil Pond 104" waste, and the pesticide waste. The failures were predominantly a combination of delamination and failure of the adhesive. These failures indicate that the loss in ply adhesion may be causing the loss in seam strength.

The results of the exposure of the CSPE membrane to the five wastes indicate that this CSPE sheeting tends to absorb water and wastes and some oil when exposed to wastes containing oily constituents. The effect of aging and exposure to wastes showed a modulus increase and decreases in elongation.

Another CSPE membrane was tested in a primary exposure cell where it was used to line immersion tanks containing deionized water and a 50:50 blend of deionized water and water from "Well 118." (Data are reported in Appendix I). This sheeting had a nominal thickness of 30 mils and was reinforced with 8 x 8 epi polyester scrim.

Note: A new grade of CSPE compound was introduced during the time this project was underway but was not included in any of the tests reported. This new barrier compound is significantly more resistant to swelling by different liquids. Also, there has been a shift from nylon to polyester as the reinforcing fabric (Matrecon, 1983).

Elasticized Polyolefin (ELPO) Membrane--

The elasticized polyolefin membrane (No. 36) was a black thermoplastic sheeting based on polyethylene. It contained a small amount of crystallinity and had a thickness of 22 mils, a specific gravity of 0.938, an ash content of 0.9%, a volatiles content of 0.15%, and an extractables content of 5.5%. During the course of the project, this sheeting was exposed as a primary liner to 12 different wastes or test media which included:

Acidic wastes:
  "HFL"
  "HNO_3-HF-HOAc"

Alkaline wastes:
  "Slop water"
  "Spent caustic"

Lead waste

Oily wastes:
  "Oil Pond 104"
  "Slurry oil"
  "Weed oil"

Pesticide waste

Well water, "Well 118"

"Well 118":water, 50:50

Deionized water.

The initial exposures were tested in duplicate with the following wastes:

Acidic waste, "HNO_3-HF-HOAc"

Alkaline waste, "Spent caustic"

Lead waste

Oily wastes:
  "Oil Pond 104"
  "Slurry oil"

Pesticide waste.

Subsequently, in order to furnish tanks for immersion testing, the remaining wastes were placed into exposure testing in combination with the ELPO liner. Some results of exposure to these wastes are reported in Tables 37 through 44.

The elasticized polyolefin liner had only small increases in volatiles content in those wastes that were predominantly water; for example, the pesticide, the lead, and the "Spent caustic" wastes. Exposure to those wastes that contained oily constituents, particularly with the "Oil Pond 104" waste and the "Slurry oil" waste, resulted in increased volatiles contents. There were significant increases in the extractables content of the elasticized polyolefin by the oily wastes which resulted in major drops in tensile strength and modulus and softening of the sheeting. There was also a significant increase in volatiles content by the elasticized polyolefin exposed to the acidic waste, "HNO_3-HF-HOAc" i.e. 5.3% in 1217 days and to the alkaline waste, i.e. 10.83% in 2299 days.

The seam in the elasticized polyolefin specimen was 0.5 in. in width and was made by the supplier using a heat sealing device of their design. This method of seaming was used in both factory and field. The value of original strength of the seam tested in the shear mode was 28.8 ppi with the failure at the seam edge. Almost all of the values measured after exposure were higher and all were at the seam edge. The only measurements that came in lower were taken from the specimens that had been exposed to "Oil Pond 104" waste; their strengths in shear were 19.3 and 18.3 ppi at 521 and 1357 days, respectively, reflecting the loss in the strength of the sheeting.

In the peel mode the value was 21 ppi with failure at the line of peel, which occurred catastrophically. The values of most of the exposed specimens were the same as was the type of failure. The specimens exposed to "Oil Pond 104" waste and the "Slurry oil" waste were lower, probably reflecting the loss of strength of the sheeting resulting from absorption of the oil from the wastes.

Overall, the elasticized polyolefin lining material showed good retention of properties in the aqueous wastes. However, those wastes that contained significant amounts of oil were absorbed by the liner, resulting in loss of tensile strength, modulus, and tear strength. Problems with the ELPO seams are indicated by the types of failure when tested in peel.

Note: The adverse effects of oil on this sheeting were also observed in the tub test described in Section 8.

Ethylene Propylene (EPDM) Membrane--

The EPDM rubber (No. 26) that was tested was a crosslinked sheeting of 30-mil thickness. It had a specific gravity of 1.169, an ash content of 7.67%, a volatiles content of 0.50%, and an extractables content of 22.96%. The high extractables content shows the high oil content that is common to many EPDM compounds. Two sets of specimens of this sheeting were exposed to four different wastes:

Acidic waste,"HNO_3-HF-HOAc"
Alkaline waste, "Spent caustic"
Lead waste
Pesticide waste.

The oily wastes were specifically excluded from the exposure testing based upon the preliminary compatibility testing and the oil sensitivity of this type of rubber.

The first set of test specimens was removed after 497 to 526 days of exposure and the second set after 1147 to 1344 days of exposure.

This sheeting increased in volatiles content in all the wastes, particularly on exposure to acidic waste; these volatiles were predominantly water. The extractables changed slightly, generally upward.

The 2-in. seam incorporated in the EPDM exposure specimen was fabricated by Matrecon using a two-part vulcanizable adhesive and a gum tape furnished by the supplier. The seam had a strength in the shear mode of 39 ppi with the failure in the adhesive. After exposure, the values in the shear mode were all higher except for the seam in the sample that was in contact with the lead waste, for which there was a substantial loss in value. In all except one case, the failures were in the adhesive.

In the peel mode, the adhesion was 4.9 ppi which, as in the case of the shear mode, was retained during exposure, except for the lead waste. After 499 days of exposure the adhesion was 3.5 ppi and after 1344 days it was 2.3 ppi.

Overall, the EPDM membrane was affected only moderately by the four wastes to which it was exposed. Of the four wastes, the acidic waste appears to have been the most aggressive toward the EPDM compound; the effects, however, were not large. The seam strength was low before exposure and decreased with exposure, indicating a probable inadequacy of the seaming method. This membrane was not exposed to oily wastes which would have severely swelled it.

Neoprene Membrane--

The neoprene sheeting (No. 43) that was tested was vulcanized and not fabric-reinforced. It had a nominal thickness of 34 mil, a specific gravity of 1.477, an ash content of 12.3%, a volatiles content of 0.45%, and an extractables content of 13.69%. Two sets of specimens of this membrane were exposed initially to four different wastes:

Alkaline waste, "Spent caustic"
Lead waste
Oily waste, "Oil Pond 104"
Pesticide waste.

Subsequently, it was exposed to two additional wastes in exposure tanks for the immersion testing:

Industrial waste, "Basin F"
Well water waste, "Well 118"

Because neoprene is generally considered to be an oil-resistant rubber, it was exposed to the oily wastes, as wastes of this type are aggressive to many of the lining materials.

The first set of specimens was removed from exposure after 494 to 526 days and the second set was removed after 1237 to 1356 days.

All the neoprene membrane specimens increased substantially in volatiles content in all of the wastes, increasing from 0.45% to 11.29 - 21.31%, except in the "Spent caustic," in which the values increased 4.40 to 5.67%. On the other hand, the extractables content changed little even for the specimens exposed to the oily wastes. Consequently, it appears that most of the liquid absorbed by the neoprene specimens was water. The neoprene specimens exposed to the lead waste, "Oil Pond 104" waste, and the pesticide waste softened considerably and had a low retention of S-100. This is probably the result of absorbing water. The specimens exposed to the spent caustic softened little and retained their elongation best, probably the result of the high dissolved solids content of the wastes. Low absorption of highly concentrated brines is characteristic of neoprene compounds.

The 2-in. seam incorporated in the neoprene specimen was fabricated by Matrecon with a lap sealant furnished by the supplier. The strength of the unexposed seam in the shear mode was 45.7 ppi with the failure at the bond between the adhesive and the liner surface. The effects of the exposures to the different wastes varied with the waste. In the case of the seams exposed to "Spent caustic" waste the test value for seam strength increased with the failures occurring at the clamp edge or in the sheeting. In the case of the lead waste, the strength value was less with failures both at the surface of the adhesive and in the liner itself. For the seam in the liner exposed to "Oil Pond 104" waste, some of the failure was in the adhesive but the principal failure was in the sheeting, reflecting the loss in strength due to the absorption of the waste. In the case of the seams in the specimens exposed to the pesticide waste, the failures of the test specimens were at the clamp edge or at the seam edge. The low values probably reflect the loss of strength of the sheeting.

In the peel mode, the unexposed seam had a strength of 8.2 ppi with failure at the bond between the adhesive and the liner. The failures in all of the exposed specimens were of this type; however, the peel strength of the seams increased during the exposure in the spent caustic, but decreased during the exposure in the oily and lead wastes.

The neoprene membrane (No. 43) showed considerable absorption of both water and oily constituents. The water absorption was the lowest on exposure to the "Spent caustic" waste, which is essentially a concentrated brine, a type of medium in which neoprene compounds are known to have low absorption. The effect on physical properties also varied considerably depending largely on the amount of swelling, which is indicated by the volatiles. The oil resistance normally associated with neoprene was not apparent in these tests. It appears that, if a neoprene membrane is being considered for lining a water impoundment, it should tested for compatibility with a sample of the waste to be impounded or a prototype of that waste.

Polyester Elastomer (PEL) Membrane--

The polyester elastomer membrane (No. 75) was a developmental product with a thickness of 7 mils. It was furnished by the supplier with a heat-sealed seam, because a higher temperature than we could obtain with our equipment was needed to prepare seams. This film was based on a partially crystalline polymer that melts in the 188 to 207°C range. It had a specific gravity of 1.236, a volatiles content of 0.26%, an extractables content of 2.74%, and an ash content of 0.38%. A thermogravimetric analysis of this film indicated that it contained 91% polymer, 3% plasticizer, and 6% carbon black.

The polyester elastomer sheeting was included in this project because of its reported resistance to hydrocarbons and other oily materials. As a primary liner it was exposed to six wastes:

Acidic waste,"HNO_3-HF-HOAc"

Alkaline waste, "Spent caustic"

Lead waste

Oily wastes:
"Oil Pond 104"
"Slurry Oil"

Pesticide waste.

The first set of samples was removed from exposure after 328 to 526 days and the second set was removed after 509 to 1357 days.

This was the only material in the entire program that failed by cracking and leaking on exposure to a waste, in this case, the acidic waste "HNO_3-HF-HOAc". This shows the sensitivity of this polymer to acidic materials which caused it to degrade by hydrolysis. After 323 days of exposure to this waste, it had retained only 33% of its tensile strength and lost almost all of its elongation.

With respect to exposure in the oily wastes, the polyester elastomer lost significantly in its physical properties in the "Slurry oil" waste and similarly, but not to the same degree, in the "Oil Pond 104" waste. This lining material had its best retentions in the pesticide and the "Spent caustic wastes.

The 0.5-in. wide seam incorporated in the polyester elastomer sheeting was furnished by the supplier and was made by heat sealing. The strength of the unexposed seam in the shear mode was 21.2 ppi with the failure occurring at the seam edge. The strength of the seams after exposure remained high. The test specimens broke either at the seam edge or at the clamp edge. There was some indication that the lower values where the failures occurred at the clamp edge were the result of the somewhat lower strength of the sheeting due to absorptions of oily constituents from the wastes.

The strength of the unexposed seam in peel was 20.8 ppi with failure occurring at the line of peel. On exposure to the lead, oily, and pesticide wastes, there was some reduction in strength with the breaks predominantly at the peel line.

The results show that PEL membranes should not be exposed to highly acidic wastes, but indicate that they may be useful for specific waste streams. Compatibility tests must be carried out with a sample of the waste to be contained or with a prototype of that waste.

Note: The polyester elastomer sheeting was the thinnest of all the materials tested in this project. New versions of this type of material are now available with improved properties for liner applications.

Polyvinyl Chloride (PVC) Membrane --

The specific polyvinyl liner membrane (No. 59) that was tested was a sheeting with a nominal thickness of 30 mils. It had a specific gravity of 1.280, an ash content of 6.97%, a volatiles content of 0.3%, and an extractables content of 35.86%. The high extractables, largely plasticizer, is equivalent to about 60 parts per 100 g of PVC resin. Specimens of this membrane were placed in exposure with 8 different wastes:

Acidic wastes:
  "HFL"
  "HNO_3-HF-HOAc"

Alkaline wastes:
  "Slop water"
  "Spent caustic"

Lead waste

Oily wastes:
  "Oil Pond 104"
  "Slurry oil"

Pesticide waste.

The first set of exposures were tested in duplicate with the following wastes:

Acidic waste:
  "HNO_3-HF-HOAc"

Alkaline waste:
"Spent caustic"

Lead waste

Oily waste:
"Oil Pond 104"

Pesticide waste.

Subsequently, in order to furnish tanks for immersion testing, the following wastes were placed in the exposure test in combination with the PVC membrane:

Acidic waste:
"HFL"

Alkaline waste:
"Slop water"

Oily waste:
"Slurry oil"

The exposures varied from 327 days to 1565 days.

The effects of exposure varied with the different wastes. The volatiles increased for all of the exposed specimens except for the specimen that was in contact with the "Slurry oil" waste. In most cases, the amount did not increase greatly after the initial exposure time. However, the increase was substantially greater for the specimens exposed to "Slop water" and the strong acid, "HNO_3-HF-HOAc." The extractables of the exposed specimen varied considerably; all, however, tended to be lower than the original value, indicating loss of plasticizer. The specimen exposed to the "Slop water" had the lowest extractables content, indicating a major loss of plasticizer. The specimens in the acidic waste also had a significant loss, although not as large as the specimens exposed to the "Slop water." Specimens in the "Spent caustic" and the pesticide wastes changed little. However, the extractables content of the specimens exposed to the lead waste and to "Oil Pond 104" waste dropped during exposure, indicating some loss of the plasticizer to the oily wastes. The effect upon physical properties was more severe. The specimens that had been exposed to the "Slop water" lost almost all of their elongation and became very hard. The specimen that had been in contact with the acidic waste, "HNO_3-HF-HOAc," lost in elongation and more than doubled in modulus S-100. Also, the specimens exposed to "Oil Pond 104" waste and the weaker acidic waste, "HFL," increased in modulus.

The 2-in. seam incorporated in the PVC specimens was fabricated by Matrecon by solvent welding using a solvent consisting of two parts or tetrahydrofuran and one part of cyclohexanone. The strength of the seam in shear was greater than 69 ppi with the failure occurring at the clamp edge. On exposure the failures were predominantly in the seam edges although some occurred outside the seam, but not at the clamp. The values on exposure were erratic; some values fell to 45 ppi and others rose to 77 ppi, more or less reflecting the strength of the sheeting itself. No failures occurred within the seams.

In the peel mode the original value for the unexposed seam was 15.2 ppi, with the bond failure between the liner surfaces. Again, on exposure, the results varied considerably, ranging from 9.8 ppi to 34 ppi. In almost all cases, the peel strength of the seams increased.

Exposure to the alkaline "Slop water" waste resulted in a major loss of plasticizer and a large increase in the volatiles content which resulted in a major stiffening and embrittlement of the liner. The elongation dropped to 3% of the original value after 1565 days of exposure and the extractables dropped to 10.4%. The second alkaline waste, "Spent caustic", did not have such an adverse effect. Exposure to the acidic wastes caused hardening of the materials and some loss in elongation, but, even after 1565 days, the effects were not large. The effects of the exposures to the oily wastes varied with the individual waste. The "Slurry oil" waste affected the sheeting only modestly during 327 days of exposure. However, the membrane exposed to "Oil Pond 104" waste showed increases in modulus, reduction in elongation, and some hardening; in the latter oily waste the extractables content was reduced, indicating that some of the plasticizer was lost during exposure.

The PVC membrane showed considerable variation in its response to the different wastes to which it was exposed. The variation was largely related to the amount of swell and the loss of plasticizer that took place during the exposure. This indicates the necessity of performing compatibility tests of the PVC membrane being considered with the waste it would impound.

An additional PVC sheeting (No. 92) was tested in the primary exposure cells where it was used to line two cells that served as immersion tanks. These cells contained deionized water and a 50:50 blend of deionized water and wellwater from "Well 118." This PVC sheeting was unreinforced and had a nominal thickness of 20 mils. (Data are presented in Appendix I). The effects of the exposure for approximately 980 days in both media were modest. This membrane softened after somewhat in DI water but hardened somewhat in the 50:50 "Well 118":DI water blend.

SECTION 7

IMMERSION TESTING OF POLYMERIC MEMBRANE LINERS

Only a limited number of combinations of polymeric membranes and wastes could be included in the primary exposure testing program. However, many other polymeric membranes were available and other potential lining materials were becoming available. These materials included membranes based on new polymers and on the polymers selected for the primary exposure program but of significantly different composition. It was desirable to expand the scope of the project to include a wider range of polymeric membranes in the waste liquids available. Furthermore, one of the objectives of this project was to develop testing procedures which could be used to assess lining materials, particularly for compatibility with waste streams.

In rubber and plastics technology, the compatibility of polymeric products being considered for service with a solvent or liquid is commonly tested by immersing samples of the rubber or plastic compound in that solvent or liquid. In this type of testing the changes in weight, dimensions, and physical properties can be used to monitor the effects of immersion. It is, of course, desirable that no changes in the material occur during service; therefore, changes in dimensions and in properties can indicate a degree of incompatibility. In some applications, specific changes in properties of the material limit the serviceability of a product. For example, a fluid delivered by a rubber hose may cause excessive shrinkage which could stiffen the hose, or swelling which could restrict the flow in the hose to such an extent that the hose would no longer be serviceable. Waste liquids can cause changes in the dimensions as well as changes in the physical properties of polymeric lining materials; therefore, measuring changes in dimensions and physical properties should be satisfactory for measuring the compatibility of polymeric lining materials.

In the initial selection of combinations of liners and wastes for long-term exposure testing, the combinations were screened by immersing small strips of the liners in the different wastes. It was felt that short-term tests of a few weeks or less would indicate those combinations which were incompatible and should not be included in long-term exposure. This testing quickly eliminated several combinations; however, longer term tests were needed to reflect the effects of extended exposure. Prolonged immersion tests had been run in assessing liners for municipal solid waste liners (Haxo et al, 1982). The results of these immersion tests correlated well with the effects of exposure in the municipal landfill simulators.

The combinations of wastes and liners selected for immersion testing in this project included combinations already being tested in the primary cells plus combinations of additional liners and wastes that had become available. Including some of the same combinations in both the immersion and the primary exposure was desirable for developing a correlation between the two-sided exposure in immersion testing and the single-sided exposure that took place in the primary cells.

The procedure followed for the immersion tests in this project is described in the following subsection.

DESCRIPTION OF METHOD OF IMMERSION TESTING

Although the exact method that was followed in immersion testing changed somewhat during the course of this project, the basic procedure was as follows:

> The exposure specimens, approximately 8 x 6 in., were totally immersed in the tanks of the primary cells, which held about 8 gal of waste, for two time periods, the longer period ranging up to several years. These slabs were preweighed and their dimensions measured. The slabs were then hung in the waste fluids with either stainless steel wire or polypropylene cord. A sufficiently large sample was selected so that physical test specimens could be cut in both the machine and transverse directions, as discussed below. The grain of the first set of specimens that was immersed was across the width; that is, the machine direction ran in the short dimension of the specimens. In later testing the grain was oriented in the lengthwise direction in which the specimens were hung.

Since the test was primarily concerned with the durability and performance of the barrier component of the liner, unreinforced (no fabric) materials were generally selected for this program. Nevertheless, two fabric-reinforced materials were included because the barrier compositions were not available without fabric at the time the project started; one of the two was a lining material included in the primary exposure. Two specimens of each lining material were immersed in each waste so that exposure data could be obtained for two time periods.

TESTING OF IMMERSED SLABS

The initial tests of the materials to be immersed were performed in accordance with ASTM test methods, i.e. the full number of replications with full-size test specimens were included in all of the tests. Results of these tests are reported in Appendixes F and G. However, in measuring the properties of the materials after immersion, a reduced number of specimens were tested for some properties and, in the case of the tensile testing, a special die with tab ends smaller than called for by ASTM D412 and D638 was used in preparing the test specimens (Figure 21). The reduced specimen size allowed us to optimize the amount of testing that could be performed on the immersed slabs. The testing that was performed toward the end of the project is listed in Table 45. In the early part of the project all the liners, even those that were fabric-reinforced or crystalline, were tested as if they were crosslinked rubbers or thermoplastics. Later, the crystalline and the fabric-reinforced materials were tested as shown in Table 45.

Measurements on Immersed Specimens

Before immersion the following measurements were made on each slab:

- Thickness.
- Dimensions in both machine and transverse directions.
- Weight.

During immersion all of these properties changed to varying amounts. If the liner materials absorbed waste constituents, generally all the values for the above parameters increased.

Due to the effect of the grain introduced into the materials during manufacture, change of dimensions differ between the machine and transverse directions. In some cases, shrinkage would occur in machine direction and swelling in the transverse direction.

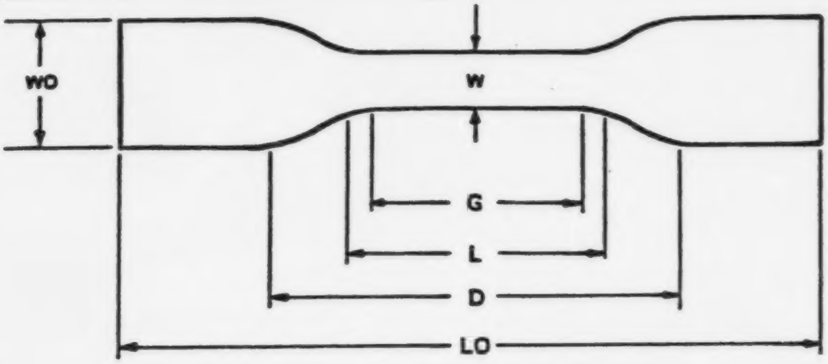


Figure 21. Special dumbbell for tensile testing of polymeric membrane specimens after immersion in waste liquids or test media. The dimensions are:

W	Width of narrow section	0.25 in.
L	Length of narrow section	1.25 in.
WO	Width overall	0.625 in.
LO	Length overall	3.50 in.
G	Gage length	1.00 in.
D	Distance between grips	2.00 in.

The width, W, of this specimen is the same (0.25 in.) as that of the Die C dumbbell in ASTM D412 and Type IV of ASTM D638. The dimension, L, in the latter dumbbell is 1.30 in. in comparison with the 1.25 in. of this dumbbell.

TABLE 45. TESTING OF POLYMERIC MEMBRANES IN THE IMMERSION TEST

Test	Type of compound and construction			
	Crosslinked or vulcanized	Thermoplastic	Crystalline	Fabric-reinforced
Measurements on immersed specimens				
Thickness, method	Dead weight gage, mils	>	>	>
Report	Percent change	>	>	>
Dimensions, method	Ruler graduated to 0.01-in.			
Report	Percent change in machine and transverse directions	>	>	>
Weight, method	Analytical balance			
Report	Percent change	>	>	>
Analytical properties				
Volatiles - method	MTM-1[a]			
Report	Moisture, % Non moisture, %	>	>	>
Extractables - method	MTM-2[b]			
Report	Solvent	>	>	>
Physical properties				
Tensile properties				
Method	ASTM D412	ASTM D638	ASTM D638	ASTM D751, Mtd B
Type of specimen	Special dumbbell[c]	Special dumbbell[c]	Special dumbbell[c]	1-in. wide strip and 2-in. "jaw" separation
Number of specimens	3 in each direction	>	>	>
Speed of test	20 ipm	20 ipm	2 ipm	12 ipm
Values to be reported	Tensile strength, psi Elongation at break, % Stress at 100 and 200% elongation, psi	Tensile strength, psi Elongation at break, % Stress at 100 and 200% elongation, psi	Tensile stress at yield, psi Elongation at yield, % Tensile strength at break, psi Elongation at break, % Stress at 100 and 200% elongation, psi	Tensile at fabric break, ppi Elongation at fabric break, % Tensile at ultimate break, ppi Elongation at ultimate break, % Stress at 100 and 200% elongation, ppi
Modulus of elasticity				
Method	...	...	ASTM D882	...
Type of specimen			Strip 0.5 x 6-in.	
Speed of test	...	...	0.2 ipm	...
Strain rate	...	...	0.1-in./in./min.	...
Tear resistance				
Method	ASTM D624	ASTM D1004	ASTM D1004	d
Type of specimen	Die C	...	...	...
Number of specimens	2 in each direction	>	>	...
Speed of test	20 ipm	20 ipm	2 ipm	...
Puncture resistance				
Method	FTMS 101B-2065	>	>	>
Type of specimen	2 x 2-in.	>	>	>
Speed of test	20 ipm	>	>	>
Number of specimens	1 or 2	>	>	>
Report	Thickness, mil	>	>	>
	Stress, lb	>	>	>
	Elongation, in.	>	>	>
Hardness, Duro (5-sec)	Duro A Duro D (if Duro A >80)	>	Duro D	Duro A Duro D (if Duro A >80)

[a] Matrecon Test Method No. 1 (Matrecon, 1983, pp. 338-39).

[b] Matrecon Test Method No. 2 (Matrecon, 1983, pp. 340-43).

[c] See Figure 21.

[d] No tear resistance test is recommended for fabric-reinforced sheetings in the immersion study.

Analytical Properties

In addition to the weight change that occurred in the test specimens, the volatiles and the extractables of the specimens were determined. Both of these properties relate to the amount of waste that is absorbed by or the components that are extracted from a liner material. Most of the volatiles are moisture, and the difference between the moisture component and the nonmoisture component was not distinguished during most of the testing.

The extractable content of a polymeric membrane can change during immersion by the absorption of nonvolatile organic constituents into the lining material and also by the extraction of parts of the original compound, e.g. plasticizers, by the waste fluid.

Physical Properties

The following physical properties of the membranes were measured after two different periods of immersion:

- Tensile properties, including tensile strength, elongation at break, and stresses at 100 and 200% elongation.
- Modulus of elasticity for crystalline type materials.
- Tear resistance for all materials except the fabric reinforced.
- Puncture resistance.
- Hardness.

The tear resistance of the fabric-reinforced membranes was not determined because the fabric in the Type C test specimens does not break but pulls out of the rubber matrix during the test. In order to get threads to break during tear testing, considerably larger specimens are required than the ASTM D624 Die C type test specimen. Specimens 8 x 6 in. are used to measure tear resistance of fabric-reinforced membranes.

Puncture resistance measured in accordance with Federal Test Method Standard 101B-2065 was particularly convenient for determining the effect of immersion. If there was sufficient material, this test was run in duplicate.

Hardness changes reflected the softening or hardening of the material during immersion. Durometer A was measured on all except the crystalline type materials and Durometer D was used in addition to Durometer A if the Durometer A reading was greater than 80 points.

The anisotropy introduced in the liner material by the grain affects the tensile and tear properties of the sheeting and, consequently, these were run in both directions as discussed in the results in this section.

SPECIFIC COMBINATIONS OF MATERIALS AND LIQUIDS PLACED IN IMMERSION TESTS

The immersion testing performed in this program was done in five groups:

- Group I consisted of 12 different polymeric membrane liners immersed in 9 different wastes. Five of the membranes were the same as those tested as liners for primary cells.
- Group II consisted of the testing of the same lining materials immersed in two important standard test media, i.e. distilled water and a 5% solution of NaCl.
- Group III added a crosslinked CPE and three crystalline materials to the program. These materials were tested in the wastes and two standard test media already in the test and an industrial waste that contained considerable heavy metals and some organics.
- Group IV consisted of all the initial 12 liners in immersion in the industrial waste.
- Group V consisted of the 16 polymeric lining materials in a saturated aqueous solution of tributyl phosphate.

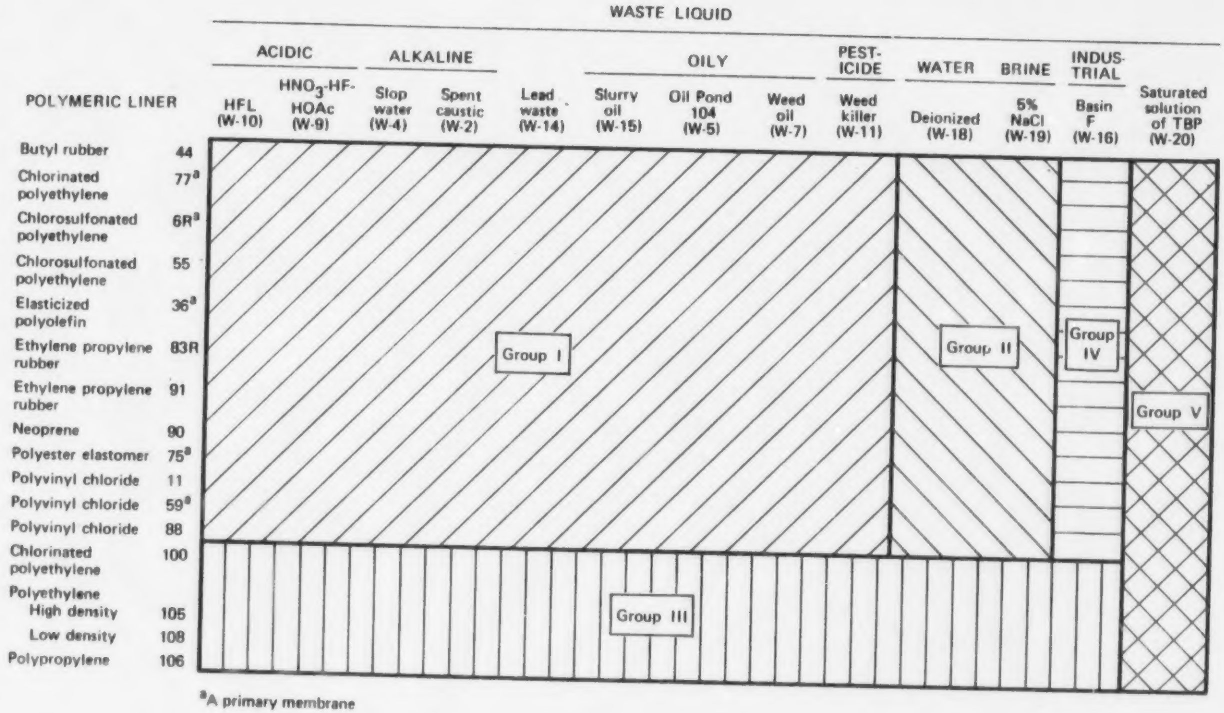
These groups are shown in the matrix given in Figure 22 and their selection is discussed in the following subsections.

First Group of Immersion Tests

In order to extend the scope of testing to other membranes and wastes and to develop correlation between immersion-type testing and the testing in the primary cells, 5 of the original lining materials, i.e. CPE (No. 77), CSPE (No. 6R), elasticized polyolefin (No. 36), polyester elastomer (No. 75), and PVC (No. 59), were placed in immersion in all of the wastes that were in the primary test and some additional wastes, i.e. a second acidic waste "HFL" (W-10), a second alkaline waste, "Slop water" (W-4), and a third oily waste, "Weed oil" (W-7). Seven additional polymeric membranes also were included.

The butyl rubber liner (No. 44) was added. Even though its 62.5-mil thickness was greater than desired, this lining material was selected because it was not fabric-reinforced. The high thread count (22 x 11 epi) of the fabric in the butyl rubber sheeting in the primary exposure testing program caused problems in evaluating the butyl itself and it was felt that a butyl material without fabric, even with its greater thickness, would be better for such evaluation.

A second CSPE (No. 55) was added. In contrast to the first CSPE liner (No. 6R), it did not have fabric reinforcement. However, as was the case with the first CSPE, it was based upon a "potable" grade CSPE compound.


WASTE LIQUID
ACIDIC
ALKALINE
OILY
PEST-ICIDE
WATER
BRINE
INDUS-TRIAL
POLYMERIC LINER
HFL (W-10)
HNO_3-HF-HOAc (W-9)
Slop water (W-4)
Spent caustic (W-2)
Lead waste (W-14)
Slurry oil (W-15)
Oil Pond 104 (W-5)
Weed oil (W-7)
Weed killer (W-11)
Deionized (W-18)
5% NaCl (W-19)
Basin F (W-16)
Saturated solution of TBP (W-20)
Butyl rubber 44
Chlorinated polyethylene 77[a]
Chlorosulfonated polyethylene 6R[a]
Chlorosulfonated polyethylene 55
Elasticized polyolefin 36[a]
Ethylene propylene rubber 83R
Ethylene propylene rubber 91
Neoprene 90
Polyester elastomer 75[a]
Polyvinyl chloride 11
Polyvinyl chloride 59[a]
Polyvinyl chloride 88
Chlorinated polyethylene 100
Polyethylene
High density 105
Low density 108
Polypropylene 106
Group I
Group II
Group III
Group IV
Group V

[a] A primary membrane

Figure 22. Groups of polymeric membrane liners placed in immersion in different wastes.

Both lining materials were produced by the same manufacturer and were approximately the same composition.

> Note: "Industrial" or low water absorption grade CSPE was not available at the time the immersion was begun.

Two EPDM membranes were added; one was a thermoplastic, fabric-reinforced sheeting (No. 83R with polyester). This was the first thermoplastic EPDM compound that we received. The second EPDM membrane was vulcanized and was 36 mils in thickness. It served as a replicate for the EPDM membrane that was being tested in the primary cells.

A neoprene (No. 90) membrane was placed in the immersion test. It had approximately the same thickness as the liner in the primary test; however, the compound differed in composition.

Two additional PVC membranes were added because of the large usage of PVC membranes as lining materials. One of the PVC's had a thickness of 30 mils and the second had a thickness of 20 mils.

Second Group of Immersion Tests

Two media that are often used in the testing of materials are deionized water and aqueous salt solutions of known concentrations, e.g. a 5% NaCl solution. Such liquids can be points of reference for many wastes. Deionized water could potentially be used in compatibility testing as a base point though in some instances deionized water can affect a polymer composition, e.g. some chlorinated polymers, more adversely than do aqueous solutions containing dissolved inorganic salts. A brine solution is of interest because of the number of wastes which contain salt. A 5% brine solution was selected as a test fluid.

All of the 12 membranes that were part of the first group were incorporated in this second group of immersion tests.

Third Group of Immersion Tests

The third group of liners and wastes consisted of a vulcanized CPE membrane (No. 100) and three partially crystalline materials, HDPE (No. 105), LDPE (No. 108), and polypropylene (No. 106) immersed in all of the 13 wastes.

The crosslinked CPE was a developmental material which was of interest because its crosslinking should make it more resistant to swelling in liquids. The three partially crystalline materials were added because of the favorable performance that crystalline materials, such as the ELPO and the polyester, had shown both in this project and in the municipal solid waste project (Haxo et al, 1983) in which LDPE and polybutylene had been studied.

Samples of these materials were obtained in 30-mil thickness from a local plastics supply firm in order to match the thickness of many of the liner materials under test. None of the three sheetings were commercial lining materials and none of them contained carbon black. At the time this facet of the work was undertaken (1977) only thin membranes (5-10 mils) of low density polyethylene were available and had been used in lining applications. Also, there was an indication that membrane liners of polypropylene had been used and that membrane liners of high-density polyethylene would become commercially available in the near future. Testing of the HDPE membrane liners that subsequently became available showed that the HDPE used in this project was considerably different in several respects from the HDPE now used in commercial linings.

These additional four membranes were immersed in the available wastes which were used for testing the first two groups of immersion tests and in an additional industrial waste that had become available and which was considerably different from the wastes that had been undergoing test. This waste ("Basin F," W-16) contained high levels of heavy metal constituents and some organics.

Fourth Group of Immersion Tests

The 12 lining materials included in the first two groups of immersion tests were placed in immersion with the industrial waste "Basin F" (W-16) in the fourth group of immersion tests. Because only one cell containing Basin F was available for the immersion, only one set of specimens of each of the 12 materials were immersed.

Fifth Group of Immersion Tests

The fifth group of immersion tests consisted of all 16 of the lining materials placed in a dilute but saturated solution of an organic chemical in water. The objective of this test was to assess the effects of a small concentration of a known organic in a water solution. The purpose for this group of exposures is discussed more fully below in the section entitled, "Immersion of Membranes in Saturated Aqueous Solutions of Tributyl Phosphate." The tributyl phosphate was selected because of its solubility in water and its relative nonvolatility. Under special circumstances it had been used as a plasticizer for PVC.

RESULTS OF IMMERSION TESTS

The results of the immersion tests are presented in a series of seven tables in Appendix J, "Exposure of Liner Specimens in Immersion Tests":

J-1. Number of Days of Immersion.

J-2. Dimensional Changes in Machine and Transverse Directions.

J-3. Percent Increase in Weight.

J-4. Percent Volatiles After Immersion.

J-5. Percent Extractables After Immersion.

J-6. Retention of Stress at 100% Elongation.

J-7. Retention of Elongation at Break.

Each of these tables include data on the effects on a single parameter of the immersion of all of the liners in all of the wastes. The data on dimensional changes are reported in percent changes in machine and transverse directions measured at the top and bottom of the immersed specimens. The data on the elongation at break and stress at 100% elongation are based upon averages of the data obtained in the machine and transverse directions. These data are reported as a percent retention of the original properties. The results of the immersion tests are discussed by polymeric membrane type.

Butyl Rubber (IIR)

Two sets of specimens of an unreinforced butyl sheeting (No. 44) were exposed in 13 different wastes or test media from 174 to 1456 days. Exposure in each waste was for two elapsed times in all of the wastes except the Basin F waste, in which only one set of specimens for most materials were immersed since only one cell of the Basin F waste was available for testing. All of the liners showed only small weight changes in the early part of the exposure; consequently, the exposure was continued in most cases for approximately 1200 days before the specimens were removed and cut for physical testing.

The specimens of the butyl membrane increased in weight during exposure from about 1% to more than 100%. The butyl specimens increased in weight in all wastes with the greatest increases in those specimens that had been immersed in the oily wastes, the aqueous solution containing tributyl phosphate, and the lead waste. The oily wastes and the lead waste caused the butyl specimens to swell significantly.

Note: Combinations testing the butyl liner with the oily wastes had been deleted from the primary tests because of the results of the preliminary compatibility study.

The changes in dimension were almost all positive and similar in both directions indicating that the strains introduced during calendering relaxed during the vulcanization of the sheeting.

The extractables contents after immersion ranged from 10.62% for the specimen immersed in the acidic waste "HFL" for 761 days to 40.34% for the specimen immersed in the "Oil Pond 104" waste for 752 days. Compared with the 11.8% value for extractables of the unexposed sheeting, these results indicate that the plasticizer in the butyl compound, usually a hydrocarbon oil, does not appear to migrate to the aqueous wastes.

"Oil Pond 104" waste caused the greatest swelling of the butyl membrane and had the greatest effect upon the membrane resulting in substantial losses of properties.

Chlorinated Polyethylene (CPE)

Two types of CPE sheeting were immersed. One (No. 77) was an unreinforced thermoplastic sheeting of 29 mil thickness and the second (No. 100) was a crosslinked CPE of 36 mil thickness. Liner 77 was also studied as a primary liner. Liner 100 was added to assess the effect of crosslinking the CPE upon the interaction of CPE membrane with wastes.

Except for a small loss in weight of the crosslinked CPE specimens immersed in slopwater and in brine (5% NaCl), all of the CPE specimens increased in weight. As indicated by the volatiles results, which were determined by heating the sheeting, much of this increase in weight was through the absorption of water.

A major difference between the thermoplastic and crosslinked CPE liners was apparent in the dimensional changes that took place during immersion. The crosslinked CPE (No. 100) increased in dimensions approximately equally in the machine and the transverse directions whereas the thermoplastic CPE (No. 77) increased more in the transverse direction and in some cases simultaneously shrank in the machine direction and expanded in the transverse direction.

As was anticipated, the crosslinked CPE increased less in weight on immersion in the wastes than did the thermoplastic uncrosslinked sheeting. However, the effects of swelling did not necessarily carry over into the physical properties as can be seen by an inspection of the data in Appendix J-6 (Retention of Stress at 100% Elongation). In the case of the samples in the nonoily wastes, both the crosslinked and the thermoplastic sheetings tended to increase in modulus (S-100), i.e. to stiffen, with exception of the crosslinked CPE immersed in the acidic wastes. The thermoplastic CPE tended to stiffen more than the crosslinked CPE. Of the samples immersed in the oily wastes, even though both sheetings lost in modulus, the retention of S-100 was significantly less for the thermoplastic CPE than for the crosslinked. The thermoplastic CPE lost severely on exposure to the "Slurry oil" waste, the lead waste, the "Weed oil" waste, and the trace organic solution. The "Weed oil" waste appeared to have completely dissolved the thermoplastic CPE specimen. The retention of elongation at break was generally less than 100% and was about equal for the thermoplastic and crosslinked compounds.

Chlorosulfonated Polyethylene (CSPE)

Two versions of a CSPE lining material produced by the same manufacturer were immersed in the 13 wastes or test media for two time periods, the shorter time period ranging from 174 to 522 days and the longer from 751 to 1456 days. One membrane was a fabric-reinforced sheeting (No. 6R) which had been previously tested in the study of lining materials in contact with MSW leachate (Haxo et al, 1982) and as a liner for the primary exposure test cells in this project. The second was an unreinforced CSPE sheeting (No. 55) of 35-mil thickness. Both were "potable" grade CSPE compounds.

Overall, the two CSPE materials responded very similarly to the wastes, even though one contained fabric reinforcement and the other did not. All of the specimens in immersion swelled and gained in weight, with the samples immersed in the oily wastes increasing the most. These increases ranged from 30% to more than 350%. In the predominantly aqueous wastes, the weight increases and the amount of water absorbed as indicated by the volatiles were less significant. Furthermore, the weight increases of the specimens immersed in deionized water were greater than those immersed in wastes and liquids containing high salt concentration, e.g. the "Spent caustic" waste. Analysis for extractables indicated that the extractables increased only among those specimens immersed in the oily wastes. The specimens immersed in aqueous wastes had approximately the same extractables as the unexposed sheeting. This indicates that the loss of plasticizer to the waste is low.

The "Weed oil" waste was by far the most aggressive toward this lining material resulting in significant losses in modulus. Losses in modulus (S-100) were much less in the other oils and not to the extent that might be anticipated from the swelling. These materials appear to have crosslinked during the immersion.

With respect to the other nonoily wastes, the increase in weight ranged from approximately 2.2 to 20%, with major increases in stiffness, i.e. S-100, and in some cases loss in elongation, such as in the slopwater. Again, this reflects the crosslinking reaction that took place during the immersion.

These data generally indicate the need for compatibility testing of CSPE liners in wastes with which they might be used and that long exposures are probably necessary for the proper assessment of compatibility. Again, as indicated above, both of these CSPE sheetings were based on potable grade CSPE compound. The more recently developed industrial grades should show significantly less adverse effects on exposure to wastes such as were used in this project.

Elasticized Polyolefin (ELPO)

Two sets of specimens of a single elasticized polyolefin sheeting (No. 36) was immersed in the 13 wastes and test fluids for two time periods. The shorter time period ranged from 174 to 522 days, and the longer from 751 to 1456 days (Appendix J-1).

Almost all specimens gained some weight; those specimens immersed in the aqueous liquids tended to gain the least and those immersed in the oily wastes gained the most. Those specimens immersed in the saturated tributyl phosphate solution had intermediate gains in weight. Among the nonoily wastes, the slopwater, which was a highly alkaline material, caused a substantial weight increase in this sheeting. These weight increases were reflected in the retention of stress at 100% elongation (S-100). The specimens that increased in weight tended to soften, i.e. had lower retention values. The retention of elongation at break varied more but in most cases the retention was lower with the more swollen specimens.

The main conclusion to be drawn from the immersion tests of the elasticized polyolefin is that this sheeting should not be used in contact with hydrocarbon oily wastes, with wastes that are highly alkaline and possibly those wastes that are highly acidic. Also, if this membrane is being considered as a lining of an impoundment of waste liquids, its compatibility with the waste over an extended period of time should be determined.

Ethylene Propylene Rubber (EPDM)

Two sets of specimens of two sheetings were immersed in the 13 waste liquids and test fluids for two time periods. The shorter time period ranged from 174 to 522 days and the longer period from 751 to 1456 days. One sheeting was crosslinked EPDM (No. 91) and the second was a fabric-reinforced (8 x 8, polyester) thermoplastic sheeting (No. 83R).

The response of these sheetings to the wastes was generally different though in some "wastes", e.g. in deionized water, the two responded similarly.

Contrary to what would normally be expected, the thermoplastic EPDM sheeting (No. 83R) absorbed less waste and retained its properties better than the crosslinked EPDM sheeting (No. 91). Even in the case of the oily wastes, the absorption was generally less.

In the case of the nonoily wastes, the crosslinked EPDM increased significantly more in weight in the acidic wastes and in the "Weed killer" waste. This appears to reflect the sensitivity to moisture on the part of this sheeting.

The volatiles after immersion indicate that, in most cases, the weight increases were due to water absorption, particularly in the crosslinked specimens immersed in the acidic wastes. The extractables data indicate that little if any of the plasticizer in the original compounds was lost to the waste. The only specimens that increased in extractables were those that had been immersed in the oily wastes.

The effects of the immersion on stress at 100% elongation generally reflected the amount of weight increase, particularly for those specimens immersed in the oily wastes and the crosslinked EPDM immersed in the "HNO_3-HF-HOAc" acidic waste.

The effects of the immersion on elongation were highly variable with retentions varying from 47 to 154%. The elongation of the thermoplastic, fabric-reinforced sheeting increased in all cases, whereas most of the crosslinked specimens immersed in the oily wastes lost in elongation.

Neoprene (CR)

Two sets of specimens from one neoprene liner (No. 90) were immersed in the 13 wastes and test liquids from 174 to 1456 days. All of the specimens gained weight on immersion and the weight gains were the greatest in the oily wastes. The smallest weight increases occurred in those wastes that had a high content of dissolved inorganic salts, such as the brine and the alkaline wastes. The intermediate weight losses occurred in specimens exposed in those wastes that were predominantly water with low dissolved salts or acids, for example, the pesticide and "HFL" wastes. As is characteristic with neoprene compounds, the specimens in solutions containing dissolved inorganic salts swelled less than the specimens immersed in deionized water.

The effects upon the stress at 100% elongation more or less followed the increases in weight, that is, the swelling of the neoprene specimens. The losses in modulus were the greatest in those specimens exposed to the oily wastes. On the other hand, the neoprene specimens retained less than 100% of their original elongation at break on exposure to all the wastes, with values ranging from 47 to 98%. The magnitude of retention varied considerably and did not follow the weight increases.

Overall, this neoprene membrane was sensitive to the oils and to water. Since, in most cases, the effect increased with time, low retentions may occur on extended immersions. This particular neoprene compound contained a high level of extractables (21.5%) which may have adversely affected its performance in these tests.

Polyester Elastomer (PEL)

Two sets of specimens of the experimental polyester elastomer film (No. 75) were immersed in the 13 wastes and test liquids. The specimens increased in weight on immersion in all but possibly two of the wastes. These losses were minor and may have been within experimental error. The polyester elastomer specimens gained weight principally in the hydrocarbon oily wastes; the gain in weight in the solution containing tributyl phosphate was not as great. The specimens immersed in the acidic waste "HNO_3-HF-HOAc" gained weight significantly; in addition, they lost severely in elongation as had the same sheeting in the primary cells. This deterioration was the result of the hydrolysis of the polyester polymer.

The retention in stress at 100% elongation of the polyester elastomer film, tended to be inverse of the increases in weight; that is, the modulus retention decreased with the increase in weight. Except for the loss in elongation in the acidic waste, which was drastic, the retention of elongation was good.

Note: This membrane did not have as good resistance to oil as had been indicated by the supplier. However, according to the supplier, new versions are now available with improved properties.

Polyethylene (PE)

Neither the high- nor the low-density polyethylene immersed in this study were commercial lining materials and both were nonpigmented. Both sheetings exhibited comparatively low values for weight increases in all wastes. However, as with the other polymeric membranes, the weight increases, though low, were the greatest in the specimens exposed in the oily hydrocarbon wastes in which the low-density polyethylene sheeting increased in weight more than the high-density polyethylene. The saturated tributyl phosphate solution caused only a slight increase in weight, that is 0.3 to 0.5%. The weight increases in all the other wastes were less except for the low-density polyethylene exposed to "Slopwater" waste.

For the first set of exposed polyethylene samples, the tensile tests were run at 20 inches-per-minute (ipm) such as are performed on rubber compounds; however, this speed was not standard for the polyethylenes and, consequently, the second set was run at 2 ipm and the tensile properties on the unexposed membranes were redetermined at 2 ipm. The retention of elongation of the high-density polyethylene sheeting on the longer exposure periods varied considerably; for example, some major losses in elongation occurred in the samples that had been exposed to the acidic waste "HNO_3-HF-HOAc". These major losses in elongation reflect the sensitivity of the specific polyethylene to specific wastes.

> Note: As indicated above, the sample of high-density polyethylene (No. 105) investigated in the immersion test was not a commercial lining material, nor was the grade of HDPE known to have been used in the manufacture of membrane liners. We later found by differential scanning calorimetry that it was considerably more crystalline than grades of polyethylenes used in lining materials, had a higher density, and a considerably higher modulus of elasticity than the modulus of the HDPE used in liners that are now available. We also found that the HDPE (No. 105) showed indications of inadequate resistance to environmental stress-cracking compared with the HDPE currently used in liners. In a stress-cracking test, the HDPE sheeting (No. 105) was tested in accordance with ASTM D1693 but at a thickness of 30 mils, which is below the required thickness for this test. It sustained some early breaks (216 h) at 100°C, whereas currently-available HDPE liners tested at thicknesses of 80 and 100 mils did not fail in 1,000 hours at 100°C. At the present state of knowledge of HDPE liner performance, specific correlations between environmental stress-cracking resistance under different conditions and field service of liners have not been established.

In the immersion test, the polyethylenes showed low weight increases in all of the wastes and fluids. The low retention of elongation of the particular HDPE that was tested indicates the need for carefully selecting the type of HDPE to be used in the manufacturing of liners for waste impoundment facilities. The data show that some grades of HDPE are probably inadequate for use in liners.

Polypropylene

The polypropylene (No. 106) studied in this immersion test also was not a liner material, but, as indicated in the discussion of immersion combinations, it was a nonpigmented propylene sheeting purchased from a local plastics supply firm. In the initial tensile testing, it was treated as a rubber and tested according to ASTM D412 at 20 ipm. In subsequent tests, the rate of testing was reduced to 2 ipm. During the immersions the weight increases were small and generally less than those of polyethylene. As with the other sheetings, the increases were the greatest in the oily wastes (6-9% in 1250 days). In the other wastes, the changes were less than a percent and, in some cases, there were small losses in weight which may be within experimental error.

In the short-term tests in which the testing was done at 20 ipm, the elongations were all less than 100%. After the longer immersions, the tests were run at 2 ipm which yielded elongations of 650% and greater. Retention of elongation based on the unexposed materials using the 2 ipm gave results of 92 to 112%, except for the specimen immersed in the industrial waste, Basin F, in which the retention was 76% after 1287 days of exposure.

Overall, this polymeric sheeting retained its properties well after exposure to the various wastes, although the effects of the oily wastes are measurable. This material, however, has not been commercialized in the United States and none was available as a liner material for testing. The tests we performed on this non-liner propylene show that this highly crystalline polymer is quite compatible with many wastes.

> Note: This particular polypropylene sample cracked relatively early in the environmental stress cracking test, ASTM D1693; thus, this grade of polypropylene would probably not be satisfactory as a lining material.

Polyvinyl Chloride (PVC)

Three different PVC membranes were immersed in the 13 wastes and test liquids from 174 to 1456 days. The three (Numbers 11, 59, and 88) represented PVC membranes from three different suppliers and were 22, 33, and 20 mils in thickness, respectively. Liner No. 59 was tested as a primary liner in the project. These three PVC membranes differed considerably among themselves and in their responses to the different wastes.

The changes in weight during immersion ranged from significant loss, i.e. 15.7%, to substantial gains, i.e. 57.7%, depending on the waste liquid and the immersion time. All three sheetings lost weight in the slopwater, the brine, and "Oil Pond 104" waste. In water they all initially increased in weight and then lost weight. In other wastes, some gained and others lost weight. All gained in volatiles content which reflects the absorption of water from the wastes. Analysis of the extractables after removal of the volatile constituents indicates that in several cases there was substantial loss in the original plasticizer, such as was the case with the specimens immersed in slop water. In some cases, the extractables content increased due to the absorption of nonvolatile organics, e.g. hydrocarbon oils.

The effects upon the dimensions of the immersed specimen were quite varied but generally followed the weight changes, i.e. if the specimen gained weight the dimensions increased. If the specimen lost weight, it decreased in length and width. In the case of shrinkage, a confined sheeting would come under tension and a hole would become enlarged. There appeared to be a dimensional effect; the dimension in the transverse direction tended to increase more and shrink less than the dimension in the machine direction.

The effects upon properties appear to correlate reasonably well with the weight changes. Those specimens that lost weight all became harder as shown by the retention of stress at 100% elongation; those that increased in weight generally became softer. The effects of weight loss also show up in the retention of elongation for those materials that lost substantially in plasticizer and which not only became stiff, but lost in elongation.

Overall, the PVC lining materials varied considerably in their response to different wastes. Certain wastes, such as the highly alkaline wastes, are particularly aggressive toward the PVC and certain oils can cause loss of plasticizer and excessive stiffening and loss of elongation. Concurrent with these losses, the PVC sheeting can shrink and develop tension in the sheets. It is quite apparent that, if a PVC membrane is being considered for the lining of an impoundment that would contain potentially polluting waste, its compatibility with that waste should be determined.

IMMERSION OF MEMBRANES IN SATURATED AQUEOUS SOLUTIONS OF TRIBUTYL PHOSPHATE

The tendency of organic lining materials, such as asphaltic and polymeric membrane liners, to absorb dissolved organic constituents of a waste liquid, even from dilute aqueous solutions, can have a highly significant effect upon liners on long exposures. This effect was observed with the asphalt concrete liner below the lead waste which contained a low concentration of oily components. It absorbed oily constituents and became soft and mushy.

Most waste liquids contain small amounts of organics that can be slowly absorbed by a polymeric material. The amount that can be absorbed by the liner ultimately depends on the polymer, the compound, and the chemical characteristics of the organic compounds in the waste. To demonstrate this effect, a simple "waste" and "exposure" test was conducted in which two sets of specimens of each of the 16 membranes were immersed in a dilute (<0.1%) but saturated aqueous solution of tributyl phosphate (TBP) in deionized water. TBP was selected for this study, not only because of its low solubility in water, but also because of its relatively low volatility and its phosphorous content which could be used as a tracer of the TBP movement. The concentration of TBP was maintained in the water by hanging highly TBP plasticized disks of PVC in the tanks in which the specimens were immersed. The detailed results are presented in Appendixes J-1 through J-7.

The first immersion exposure was 522 days and the second ranged from 1035 to 1119 days. On removal from immersion, the following parameters, as indicated above, were measured: weight change, extractables, volatiles (water), tensile strength, elongation at break, modulus of elasticity (for crystalline materials), hardness, tear (for all but fabric-reinforced materials), and puncture resistance.

The thermoplastics, i.e. PVC, CPE, and CSPE, increased significantly in weight and lost considerably in tensile strength and hardness. The vulcanized CPE sustained less change than the thermoplastic CPE composition. The neoprene liner, though vulcanized, swelled considerably and lost in tensile strength and hardness. The butyl and EPDM liners, also vulcanized, swelled to some extent and sustained only modest losses in properties.

The increase in weight of the samples during exposure in the saturated solution of TBP is made up of water and dissolved TBP. The total is the net of the water and TBP that are absorbed minus the plasticizer in the original compounds that may be lost to the solution.

The question remained as to how much of the original plasticizer migrates into the solution and is lost by the liner. To answer this question, analyses of two exposed liners were performed, in which the extractables obtained after the removal of water from the liner were analyzed for total TBP content, and calculations were made of the plasticizer remaining in the exposed liner. The results are shown in Table 46. These results indicate that the original plasticizer in both of these sheetings was retained during exposure and that the entire increase in extractables was a result of the absorption of the TBP.

Inspection of the data in Appendix J-3 regarding the immersion of the liner samples in saturated TBP indicate that the increase in weight tends to level off in most of the specimens. It thus appears that a maximum value of swelling may be achieved for a given organic chemical. However, these results are not confirmed by the data on volatiles (Appendix J-4) which tend to show that the volatiles increase with time.

Of particular interest are the low swelling and small losses in the values of the physical properties incurred by the semicrystalline polymers, thus showing the resistance of these materials to the absorption of an organic chemical such as TBP during the exposure to waste liquids containing minor amounts of organics.

Neither of the two polyethylene sheetings nor the polypropylene sheeting tested in this study were commercial liner membranes, but two semicrystalline polymers, HDPE and LDPE, are used currently in the manufacture of liner membranes. These three semicrystalline sheetings were tested so that comparisons could be made with other polymeric materials of the same thickness.

TABLE 46. ANALYSES OF CHLORINATED POLYETHYLENE AND POLYVINYL CHLORIDE MEMBRANES EXPOSED IN SATURATED TBP[a] SOLUTION

	Chlorinated polyethylene	Polyvinyl chloride
Liner number	77	59
Extractables of unexposed liner, % by weight of the original liner	9.13	35.9,37.4
Exposure time, days	1112	1055
Extractables after exposure, % exposed membrane after volatiles were removed[b]	26.1	51.7
TBP[c], % exposed membrane (dvb[d])	19.8	23.3
Extractables, TBP, %	6.3	28.4
Calculated plasticizer in original sheeting, % based on polymer content and plasticizer	9.4	36.4

[a] Tributyl phosphate.

[b] Specimens previously tested and held in sample storage file.

[c] Analyses of extract by gas chromatography.

[d] dvb = devolatilized basis.

SECTION 8

OUTDOOR EXPOSURE TESTS OF POLYMERIC MEMBRANE LINERS

INTRODUCTION

In many actual waste storage facilities it is desirable to leave the membranes uncovered, that is, without soil covers. Consequently, in addition to being exposed to the impounded liquid or sludge, part of the membrane, such as that on a berm, may be exposed to weathering. In such situations, the lining material is exposed to ultraviolet light, heat of the sun, wind, ozone, fluctuations in temperature, and to rain, hail, and ice. These conditions can cause some polymeric materials to degrade, resulting in softening or hardening and possibly in breakage of the liner. Membrane liners that are sensitive to these conditions are normally covered with soils to avoid the adverse effects of the weather.

In this project it was desired to develop information on the full characteristics of liners, including some of their basic limitations. Consequently, weather exposure tests were included in the program, although some of liners, for example polyvinyl chloride, are normally not exposed to the weather except for short durations.

It is recognized that geographic location can affect the weather conditions greatly; however, these test were undertaken to compare the materials. (Estimating service life of a material in outdoor conditions requires that tests be conducted in weather that is similar to the anticipated actual service.)

Two types of outdoor exposure tests were undertaken, both of which involved exposing samples of the membranes on the roof of the laboratory in Oakland, California. These tests were:

- Exposure of small slabs of polymeric membranes on a rack and removing them periodically to measure the effects of the exposure on properties.

- Exposure of samples of the membranes as linings of small tubs in which different wastes were loaded and maintained at more or less constant level.

These methods are described and the test results are presented in the following subsections.

ROOF RACK EXPOSURE

In the roof rack exposure, 6 x 6 in. specimens of 11 different polymeric membranes were exposed on a rack placed on the laboratory roof in Oakland, California, at a 45° angle to the south. Three specimens of each membrane material were hung on boards so that only one side was exposed to the sun. The rack with the test specimens is shown in Figure 23. The specimens were hung loosely to allow them to change dimensions freely. These 11 membranes were based upon the following polymers:

Butyl rubber, fabric-reinforced
Chlorinated polyethylene
Chlorosulfonated polyethylene, fabric-reinforced
Elasticized polyolefin
Ethylene propylene rubber (2 membranes)
Neoprene (2 membranes)
Polyester elastomer
Polyvinyl chloride (2 membranes).

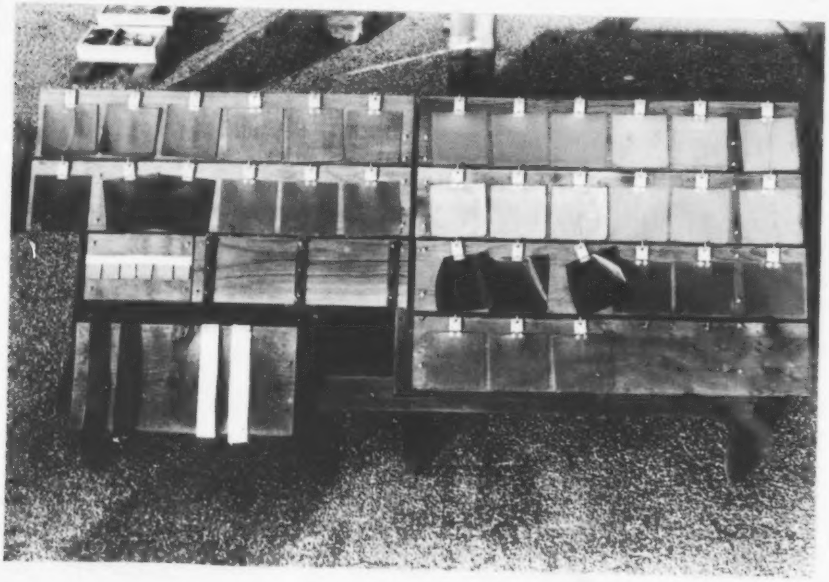


Figure 23. Exposure rack loaded with polymeric membrane specimens. The rack is exposed at a 45° angle to the south.

One specimen of each of the membranes was removed after 343, 745, and 1231 days and the following properties were determined:

Weight
Dimensions
Volatiles
Extractables

Hardness
Tensile strength
Elongation
Tear strength.

Changes in surface characteristics, including cracking and checking, were observed. Changes in these parameters and properties are reported in Appendix K.

Results

Results of 1231 days of weather exposure are presented in Table 47. They illustrate some of the differences in the weatherability of different polymeric membranes. Some of the major effects of the exposure were:

1. With only a few exceptions, the specimens lost weight and extractables content; the largest losses were sustained by the two PVC liners which lost plasticizer. The CSPE increased in weight, perhaps due to moisture absorption. The polyester elastomer membrane lost significantly in weight and, at the same time, increased in extractables content which may indicate some degradation of the polymer.

2. The moduli of all membranes increased. The amount and mechanism of increase varied with the individual membrane. The CSPE and PVC liners increased the most, and one EPDM, the elasticized polyolefin, and the polyester increased the least. The increase in modulus by the CSPE membrane was the result of crosslinking which took place during the exposure period; on the other hand, the increases in the moduli of the PVC membranes were due to loss of plasticizer as shown by the loss in weight and the reduced extractables content.

3. All membranes except that of butyl rubber lost in elongation. The butyl rubber membrane was reinforced with a fabric which controlled the elongation at break. The CSPE and neoprene membranes sustained the greatest losses.

4. The surfaces of all of the membranes changed little during the exposure, except for the PVC which crazed or "alligatored". Obviously, thinner PVC sheeting would have been seriously affected by the exposure of 1231 days.

TUB TEST

The second type of outdoor exposure test was the tub test in which small tubs lined with different polymeric membranes and partially filled with wastes were exposed to the weather. The objective of this test was to test polymeric membrane liners under conditions that simulated some of the conditions that exist in an open lined impoundment in which the liner is not covered with soil. The effects of exposure to sun, temperature

TABLE 47. OUTDOOR EXPOSURE OF POLYMERIC MEMBRANE LINERS ON ROOF RACK IN OAKLAND, CALIFORNIA FOR 1231 DAYS

Liner data				Unexposed membrane			Exposed membrane			
									Retention of property[a], %	
Polymer	Number[b]	Thickness, mil	Compound type[c]	Extractables, %	S-100[a], psi	Elongation at break[a], %	Weight change, %	Extractables, %	S-100	Elongation at break
Butyl rubber	57R	34	XL	6.4	d	42	-3.32	5.7	d	102
Chlorinated polyethylene	77	29	TP	9.1	1180	403	-3.15	6.3	139	81
Chlorosulfonated polyethylene	6R	31	TP	3.8	938	243	+1.80	3.3	248	52
Elasticized polyolefin	36	22	CX	5.5	932	665	-1.93	5.3	119	95
Ethylene propylene rubber	8	62.5	XL	23.4	335	510	-3.91	21.3	142	91
Ethylene propylene rubber	26	30	XL	23.0	358	450	-3.11	21.8	119	94
Neoprene	43	34	XL	13.7	460	320	-3.11	9.9	184	63
Neoprene	82	60	XL	13.4	383	400	-1.31	11.5	188	63
Polyester elastomer	75	7	CX	2.7	2585	575	-6.23	3.9	110	83
Polyvinyl chloride	11	30	TP	33.9	1420	357	-15.61	26.3	161	77
Polyvinyl chloride	59	33	TP	35.9	995	375	-10.31	27.8	185	86

[a] Properties of the lining materials measured in both machine and transverse directions and reported as an average or based on the average.

[b] Matrecon identification number; R = fabric-reinforced.

[c] XL = crosslinked or vulcanized; TP = thermoplastic; CX = crystalline.

[d] Specimen broke at less than 100%.

changes, ozone, and other weather factors can be assessed as well as the effect of a given waste on a specific liner. The fluctuation in the level of the waste is particularly significant in that an area of the liner is subjected to both the effects of the waste and the weather. Generally, this alternating of conditions is especially harsh on a polymeric liner material, but such conditions usually are encountered in the field in open ponds where the liners are not covered with soil.

Description of the Tubs

The tubs were constructed of 0.75 in. exterior grade plywood with sides sloping outward at a 1:2 slope. The opening at the top was 20 x 25 in. and on the inside base was 7.13 x 12 in. The depth of the tub was 10 in. A sketch of the tub is shown in Figure 24 and a photograph of six of the tubs in a catch basin is shown in Figure 25.

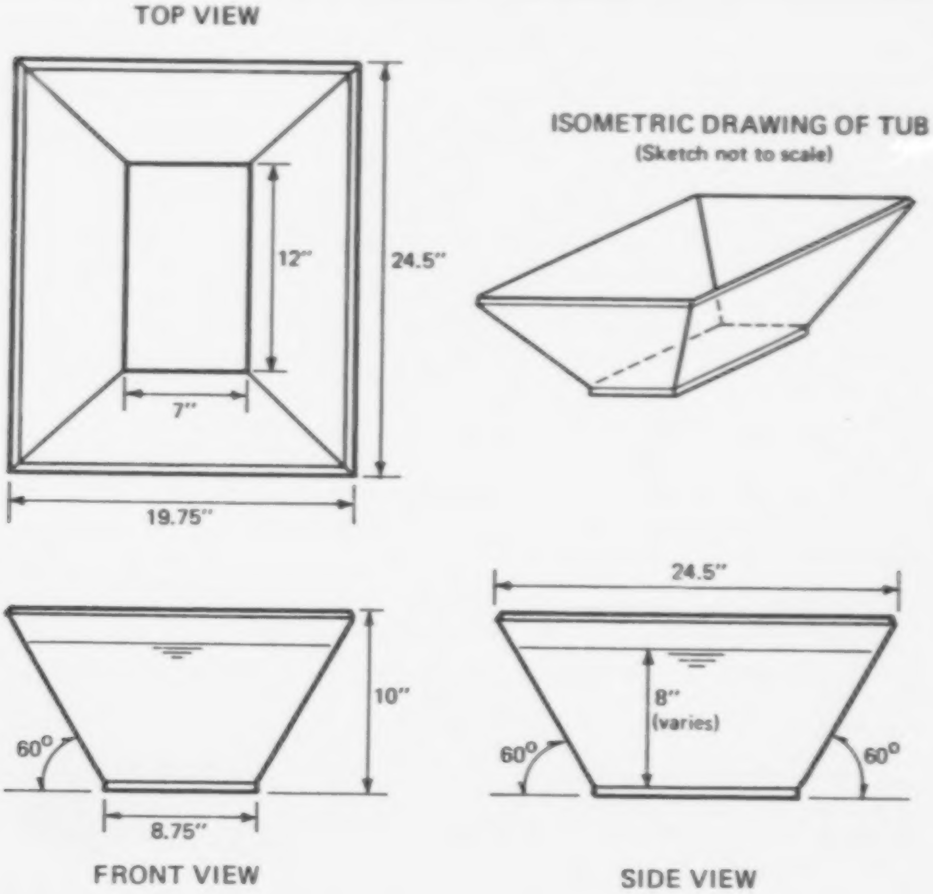

TOP VIEW
12"
24.5"
7"
19.75"
ISOMETRIC DRAWING OF TUB
(Sketch not to scale)
24.5"
10"
60°
8.75"
8"
(varies)
60°
60°
FRONT VIEW
SIDE VIEW

Figure 24. Drawing of the tub used in the roof exposure of polymeric membrane liners in contact with wastes.

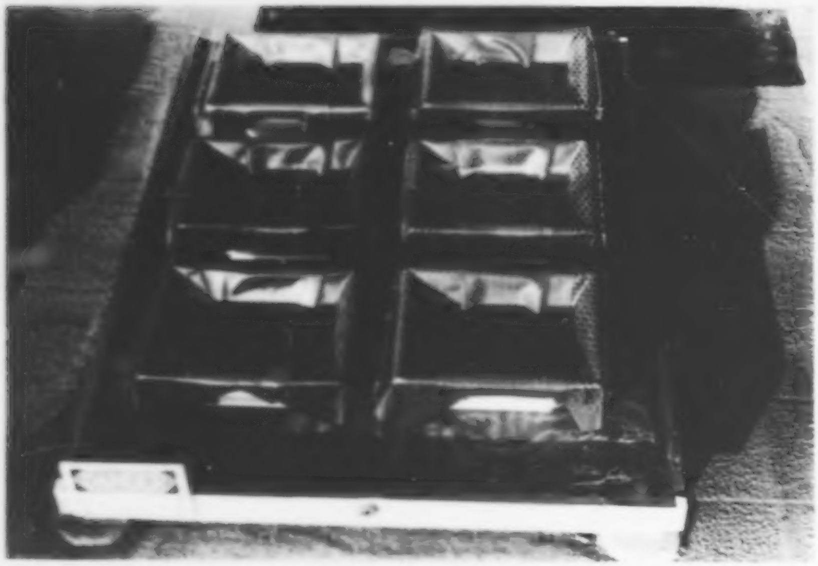


Figure 25. The open exposure tubs lined with polymeric membranes and partially filled with hazardous wastes. They were covered with chicken wire and placed in a lined shallow basin.

Test Procedure

The combinations of the liner specimens and wastes that were selected for this test are shown in Table 48. The test specimens consisted of a sheet 40 x 48 in. which, in most cases, incorporated a seam through the center. In this way the seam durability as well as that of the liner was assessed. The test specimens were draped over the tubs and folded to fit the inside corners and edges of the tubs; the excess material was allowed to hang freely over the edges. In this manner, there were a considerable number of folds and sharp angles to which the liners were exposed, particularly over the corners of the tubs. If the membrane was sensitive to the waste or to the ozone, cracking or crazing would develop.

The tubs were filled from 3/4 to 7/8 full with wastes. The liquid level was allowed to fluctuate about 4 in. Approximately 4.5 gallons of waste was required to fill a tub.

Monitoring

During the exposure the tub liners were regularly inspected visually for cracking, opening of seams, and other forms of liner deterioration. During the rainy periods, the tubs were covered. The waste levels and temperatures were measured and recorded at regular intervals. Water was added when levels became too low.

TABLE 48. COMBINATIONS OF POLYMERIC LINERS AND WASTES PLACED IN TUB TEST

		Waste identification			
		Acidic	Alkaline		Oily
Polymer	Number[a]	"HNO_3-HF-HOAc" (W9)[b]	"Slop-water" (W4)[b]	"Spent caustic" (W2)[b]	"Oil Pond 104" (W5)[b]
Butyl rubber	57R	-	X	-	-
Chlorinated polyethylene	77	-	-	X	-
Chlorosulfonated polyethylene	6R	X	X	-	-
Elasticized polyolefin	36	-	-	X	X
Ethylene propylene rubber	8	X	-	X	-
Neoprene	82	-	-	-	X
Polyester elastomer	75	-	-	-	X
Polyvinyl chloride	11	X	-	-	-
	59	-	-	-	X

[a] Matrecon liner serial number; R = fabric-reinforced.

[b] Matrecon waste serial number.

The shallowness of the tubs and the dark color of the liners resulted in high heat absorption when the tubs were exposed to sunlight; the liners and the wastes were quite warm on sunny days. The temperature was monitored regularly and the high and low temperatures in the different cells are shown in Table 49.

During much of the year, the oily wastes accumulated water (from dew) on the bottom of the tubs and did not evaporate significantly. Water, or actually an oil-water mixture, had to be pumped from the bottom of the tubs to maintain liquid levels and prevent overflows of the waste. The oil-water mixture was removed and analyzed for pH, electrical conductivity, percent solids, and other parameters as appropriate. During the rainy period, water in the catch basin was also monitored for pH and conductivity as a possible indication of leakage from the tubs containing highly acidic or highly alkaline wastes.

Results

Since the time that the tubs were placed in exposure in November 1976, only three tubs have been removed (June 1983) from service and the liners tested. All three were removed because of failures of the liners; two were lined with elasticized polyolefin and contained "Oil Pond 104 "waste.

TABLE 49. TEMPERATURE OF WASTES IN TUBS ON ROOF OF LABORATORY IN OAKLAND, CALIFORNIA

Tub	Date filled	Liner Polymer (No.[b])	Waste (No.[c])	At lowest observed temperature[a] Waste, °C	Date	Ambient, °C	Condition[d]	At highest observed temperature[a] Waste, °C	Date	Ambient, °C	Condition[d]
1[e]	11/2/76	Polyolefin (36)	"Oil Pond 104" (W-5)	13.1	10/14/77	12.8	STF	65.6	7/29/77	38.3	B
2	11/2/76	PVC (59)	"Oil Pond 104" (W-5)	12.8	10/14/77	12.8	STF	66.1	7/29/77	38.3	B
3	11/2/76	Polyester (75)	"Oil Pond 104" (W-5)	12.8	10/14/77	12.8	STF	65.6	7/29/77	38.3	B
4	11/2/76	CSPE (6)	Slopwater (W-4)	13.3	10/12/77	16.1	ST	50.0	7/29/77	38.3	B
5	11/2/76	Butyl (57R)	Slopwater (W-4)	13.1	10/12/77	16.1	ST	50.6	7/29/77	38.3	B
6	11/2/76	Neoprene (82)	"Oil Pond 104" (W-5)	12.8	10/14/77	12.8	STF	66.1	7/29/77	38.3	B
7	11/6/76	EPDM (8)	Spent caustic (W-2)	11.1	10/12/77	16.1	ST	51.1	7/29/77	38.3	B
8	11/6/76	Polyolefin (36)	Spent caustic (W-2)	10.8	10/12/77	16.1	ST	51.7	7/29/77	38.3	B
9	11/6/76	CPE (77)	Spent caustic (W-2)	11.1	10/12/77	16.1	ST	51.1	7/29/77	38.3	B
10	11/6/76	CSPE (6R)	HNO_3-HF-HOAc (W-9)	10.0	10/12/77	16.1	ST	43.5	6/7/77	28.6	U
11	11/6/76	EPDM (8)	HNO_3-HF-HOAc (W-9)	10.0	10/12/77	16.1	ST	44.0	6/7/77	28.6	U
12	11/6/76	PVC (11)	HNO_3-HF-HOAc (W-9)	10.0	10/12/77	16.1	ST	46.0	6/7/77	28.6	U
1[f]	4/6/78	Polyolefin (36)	"Oil Pond 104" (W-5)	15.5	5/10/78	18.3	ST	46.5	8/18/78	29.0	B

[a] Highest and lowest temperatures of the wastes observed from the times the tubs were filled until September 2, 1978.

[b] Matrecon liner serial number; R = fabric reinforced.

[c] Matrecon waste serial number.

[d] Weather condition:
ST = Still air; no breeze.
STF = Still air and foggy.
B = Breezy
U = Unknown - not recorded.

[e] Failed; dismantled on March 23, 1978.

[f] Replacement; first liner failed March 23, 1978 and was replaced.

This liner material had not been recommended for oily applications; however, it had functioned satisfactorily in preliminary compatibility tests. Both of these liners failed by cracking at folds. The third liner that had to be removed because of failure was a neoprene liner from the tub that contained the oily waste, "Oil Pond 104". The recovery and testing of each of the liners is described and discussed in the next three subsections.

Recovery and Testing of First Failed Elasticized Polyolefin Liner Exposed to "Oil Pond 104" Waste--

The elasticized polyolefin liner exposed to "Oil Pond 104" waste failed after 506 days of exposure at a crack in a fold at the interface between the air and the waste at the waste surface on the north sloping side of the tub. The liner appeared to have swelled considerably in this area. On removal from the tubs, physical tests were performed at four exposure locations:

- Under the waste at the bottom of the tub.
- In the waste-shade zone on the south side.
- In a shade zone only where the sheeting was draped over the north edge.
- In a waste-sunlight zone on the north side.

The waste-sunlight zone provided the most severe environment for the liner material.

The results of the tests made at the various locations are presented in Table 50. They are reported as retention of values of the original unexposed material.

As can be seen from the data, those portions of the liner that were in contact with the waste, and particularly at the air-waste interface, lost significantly in tensile strength and modulus. On the other hand, the portion of liner that was not in contact with the waste and was facing away from the sunlight retained properties.

Recovery and Testing of the Second Failed Elasticized Polyolefin Liner Exposed to "Oil Pond 104" Waste--

This elasticized polyolefin liner replaced the first liner (above) that had failed. It did not have a seam. The second liner failed after 1308 days of exposure in much the same fashion as the first. It cracked at a fold at the surface of the waste where the fold had been intermittently in contact with the waste.

On removal from the tub, this membrane showed considerable distortion and swelling at the air-waste interface area. Testing was performed in

TABLE 50. RESULTS OF TUB TEST OF FIRST ELASTICIZED POLYOLEFIN MEMBRANE EXPOSED TO AN OILY WASTE ("OIL POND 104") FOR 506 DAYS[a]
Effect of Location and Exposure in Tub

		% Retention of property of exposure			
Test	Original value	Under waste[b]	In waste and sun[c]	In waste and shade[d]	In shade[e]
Thickness, mil	23	113	113	113	98
Tensile strength, psi	2525	48	39	46	99
Elongation at break, %	655	90	82	84	101
Tensile set, %	445	76	72	74	100
S-100, psi	905	66	57	62	107
S-200, psi	1000	62	58	61	104
Puncture resistance, lb.	26.3	97	73	70	135
Elongation, in.	0.97	130	122	113	142

[a] From 11/2/76 to 3/23/78. Tensile data reported for transverse direction only.

[b] At bottom of tub.

[c] On the north side facing south.

[d] On the west side facing east.

[e] On the north side on draped section facing north.

accordance with the pattern shown in Figure 26. This pattern features the removal of a one-inch strip across the north-south axis of the tub. The thickness of the strip was measured along its length with a roller type gauge and the results are presented in Figure 27. This pattern also shows the location of the test specimens that were used.

The test results for the liner samples taken from the different locations are presented in Table 51 in comparison with the values for the unexposed material. Again, as with the first failed liner, the effects of the absorption of the oil are large. The specimens taken from the north interface had an extractables content of almost 33%, an increase of more than 20%. Retention of the tensile strength of the liner at the interface area on both the north and south sides was low. The change in tensile strength with the change in thickness during exposure caused by swelling or shrinkage is shown in Figure 28. The data on all of the individual tensile determinations have been included in the plot.

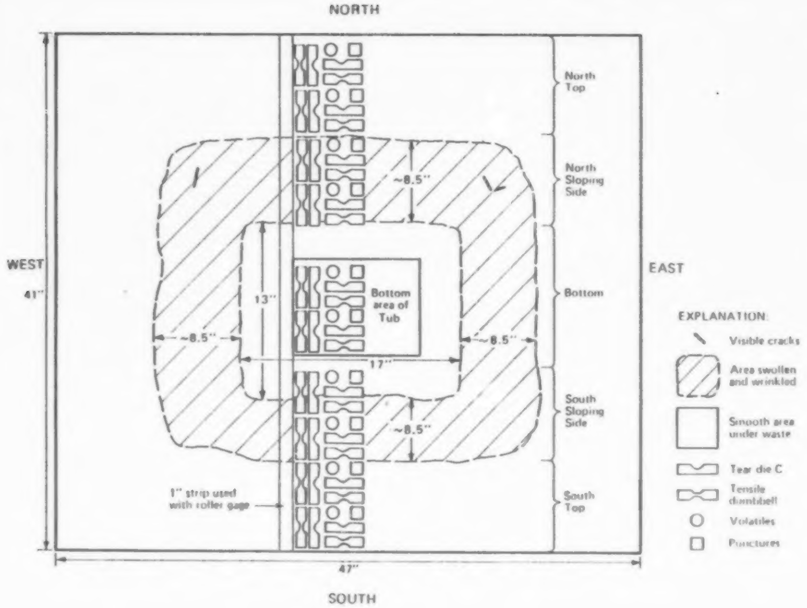

NORTH
WEST
41"
EAST
SOUTH
47"
North Top
North Sloping Side
Bottom
South Sloping Side
South Top
~8.5"
13"
17"
Bottom area of Tub
1" strip used with roller gage
EXPLANATION:
Visible cracks
Area swollen and wrinkled
Smooth area under waste
Tear die C
Tensile dumbbell
Volatiles
Punctures

Figure 26. Drawing of exposed elasticized polyolefin liner showing locations where the test specimens were cut and the orientation with respect to the tub and to the north. Location of strip for measuring thickness across specimens is also shown.

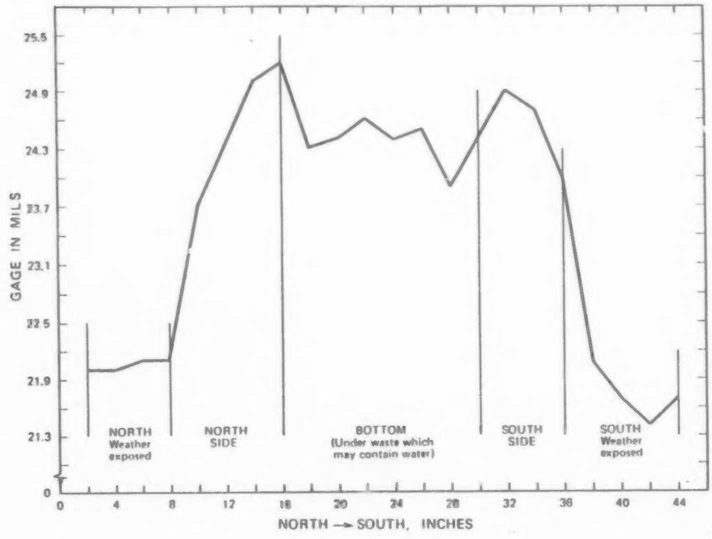


Figure 27. Thickness of strip of exposed elasticized polyolefin liner cut across the width of the liner in north-south direction.

TABLE 51. RESULTS OF TUB TEST OF SECOND ELASTICIZED POLYOLEFIN MEMBRANE EXPOSED TO AN OILY WASTE ("OIL POND 104") FOR 1308 DAYS ON LABORATORY ROOF IN OAKLAND, CALIFORNIA[a]

Variation in Location in Tub

Property	Properties of unexposed liner	Location in tub			
		North at top	North at interface	Waste only	South at interface
Analytical properties:					
Volatiles, %	0.15	1.65	6.2	8.6	8.4
Extractables, %	5.50	7.54	32.7	20.7	23.0
Physical properties:		Retention, %			
Thickness	23 mil	98	112	107	112
Tensile at break	2620 psi	84	29	48	37
Elongation at break	665 %	80	63	89	83
Tensile set	465 %	92	62	80	76
S-100	925 psi	97	49	63	59
S-200	1020 psi	95	47	61	56
Tear strength	380 ppi	94	41	56	48
Puncture resistance:					
Stress	26.3 lb	119	71	68	69
Elongation	0.97 in.	144	132	118	116

[a] From 4/6/78 to 10/1/81; tensile and tear values are averaged for both directions.

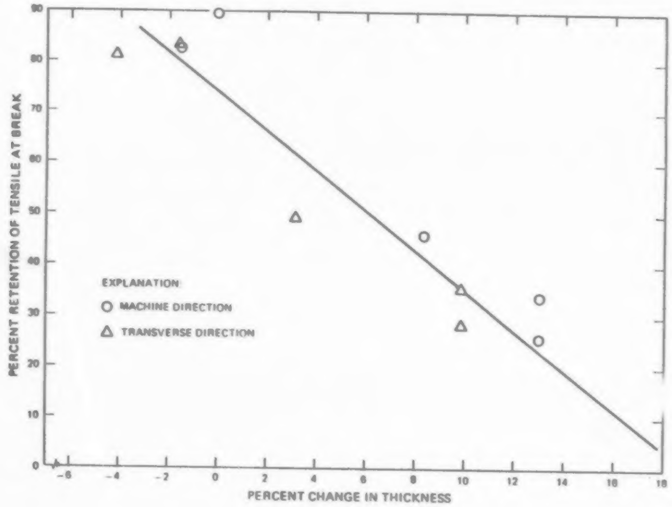


Figure 28. Retention of tensile strength of elasticized polyolefin exposed in the oily waste, "Oil Pond 104," for 1308 days, as a function of change in thickness due to swelling.

Recovery and Testing of the Neoprene Liner Exposed to "Oil Pond 104" waste--

The neoprene liner was the third to fail in the oily waste, "Oil Pond 104." It failed in the seam. The liner was removed and tested similarly to the second elasticized polyolefin. The results of this testing are shown in Table 52. The liner absorbed a significant amount of oily waste as shown by the increase in extractables in the areas where the liner was in contact with the waste.

TABLE 52. RESULTS OF THE TUB TEST OF A NEOPRENE MEMBRANE AFTER EXPOSURE TO AN OILY WASTE ("OIL POND 104") FOR 2008 DAYS ON LABORATORY ROOF IN OAKLAND, CALIFORNIA

Samples Taken From Different Locations in Tub

Property	Direction of test	Properties of unexposed liner	Position in tub				
			South top	South slope	Bottom	North slope	North top
Analytical properties							
Extractables[b], %		13.43	12.28	30.09	25.33	28.94	12.71
Volatiles Loss over desiccant at 50°C, %		...	0.6	5.1	6.5	2.1	0.5
Physical properties			Retention[c], %				
Thickness (average)		61 mil	+3	+23	+26	+23	+7
Tensile at break	Machine	1835 psi	89	51	41	31	85
	Transverse	1675 psi	89	39	45	43	92
Elongation at break	Machine	390%	71	84	79	96	74
	Transverse	410%	70	79	75	89	70
Tensile set	Machine	10%	130	90	90	80	100
	Transverse	9%	122	100	77	111	122
S-100	Machine	405 psi	152	40	29	16	154
	Transverse	360 psi	146	28	34	29	161
S-200	Machine	875 psi	135	52	44	24	132
	Transverse	705 psi	144	42	54	41	155
Tear strength	Machine	185 ppi	88	36	31	36	84
	Transverse	180 ppi	89	35	29	30	81
Puncture resistance							
Thickness		60.5 mil	+5	+26	+29	+31	+7
Stress		53.9 lb	97	54	64	32	102
Elongation		1.2 in.	51	84	81	73	68
Hardness, durometer points 5-second reading		57A	+7A	-31A	-29A	-33A	+8A

[a] Crosslinked (XL) neoprene sheeting of 60 mils thickness, without fabric reinforcement.

[b] Matrecon Test Method (Matrecon, 1983 - p. 340-343).

[c] Thickness = percent change; hardness = change in points.

Again, the value for the extractables of the sheeting from the bottom of the tub was lower than that of the sheeting on the sides at the air-waste interface area. This may be either an indication of the more severe condition at the interface or the fact that water was at times on the bottom of the tub which reduces swelling. The retention of physical properties is indirectly related to the degree of swelling by the oily waste.

There was a seam in this sheeting which was tested with the results shown in Table 53. The seam under the waste had lost severely in seam strength compared with the seam that had not been in contact with the waste.

TABLE 53. SEAM STRENGTH OF NEOPRENE LINER (#82) AFTER 2008 DAYS OF EXPOSURE IN TUB CONTAINING OILY WASTE, "OIL POND 104"

	Location in tub of sample tested		
Mode of test	East top	Bottom	West top
Shear			
Maximum, ppi	58.8	8.1	55.1
Locus of failure	AD-LS[a]	AD-LS[a]	AD-LS[a]
Peel, ppi			
Maximum[b]	9.2	1.6	8.2
Average	6.5	0.8	5.5
Locus of failure	AD-LS[a]	AD-LS[a]	AD-LS[a]

[a] AD-LS = Failure between adhesive and liner surface.

[b] Maximum is at caulked edge.

Discussion--

The results of the roof tub exposure show the importance of the location within a waste facility from which the samples are taken. The aging that occurs at the different locations in the pond can vary considerably, particularly if there is stratification of the waste which probably occurred with the oily waste. However, the most severe section was probably at the interface between the waste and the air on the north slope which faced toward the south.

SECTION 9

POUCH TEST FOR POLYMERIC MEMBRANE LINER MATERIALS

INTRODUCTION

Since the permeability of membrane liners to waste liquids and the dissolved components may not necessarily be reflected by the water vapor permeability as determined by ASTM E96, other methods of assessing the permeability of liner materials were investigated. Of these, one of the most promising was the "pouch test," in which a waste liquid is sealed into a pouch fabricated from the liner membranes under test and the pouch is then placed in deionized water that, in turn, is in a closed container. Such a condition simulates a pond containing a waste. The permeation of dissolved constituents of the waste liquid through the pouch wall can be followed by pH and electrical conductivity measurements of the water in the outer container and the permeation of water into the pouch containing waste liquid by monitoring the weight of the pouch.

To demonstrate the feasibility of the test procedure, initial experiments were carried out in the study of liners for MSW landfills (Haxo et al, 1983) on two sets of pouches fabricated from heat-sealable liner materials. One set was filled with MSW leachate and the second set with a 5% aqueous solution of sodium chloride. The liner materials in these tests included the following polymers:

- Chlorinated polyethylene.
- Chlorosulfonated polyethylene.
- Elasticized polyolefin.
- Polyester elastomer.
- Polyvinyl chloride (three different membranes).

Results are reported in the final report (Haxo et al, 1983) of that project and show the feasibility of the test for assessing simultaneously both the permeability of polymeric membranes to wastes and to some waste constituents and the effect on a lining material of one-sided exposure to a waste liquid.

In the case of test pouches containing MSW leachate, after 500 days of exposure it was apparent that there was movement through the walls of the pouches by both the water and the dissolved ingredients of the leachate.

The electrical conductivity of the water in the outer container increased indicating permeation of some ions from the leachate in the pouch into the deionized water; the odor of leachate in the outer water indicated that some organic constituents in the leachate were also permeating through the pouch walls. An increase in the weight of the pouches that contained leachate indicated movement of water through the walls into the pouches. In that series of tests, the elasticized polyolefin allowed the lowest transmission of water and dissolved components, and the chlorinated polyethylene appeared to be the most permeable.

BASIC PROCEDURE OF THE TEST

Fabrication and Filling of Pouches

The design of the experiment required that the pouches be liquid-tight so that whatever passed in or out passed through the pouch walls. This required liquid-tight seams. Heat-sealing the thermoplastic and partially crystalline membranes was the most effective method of seaming. Such seams used the least area and, if made well, appeared to hold up best. A portable heat-sealing unit, such as is used to make polyethylene bags, was used. It contained a heating element 13 in. in length.

Note: The unit was a T-Bar Plastic Sealer, Model T13 - 115v AC 130w made by Harwil Co., Santa Monica, California.

This unit was designed primarily for sealing films less than 10 mils in thickness. Consequently, some problems were encountered in seaming membranes of 30-mil thickness. Longer time, greater pressure, and higher temperatures were needed to make seams of the thicker materials. Some seams of thermoplastics, e.g. CSPE, were made with adhesives and solvents. However, adequate seams of crosslinked membranes could not be made with two-part room temperature curing adhesives and thus only pouches of thermoplastic and crystalline membranes were tested.

Figure 29 is a schematic of the pouch assembly. The dimensions of the individual pouches varied somewhat but, in most cases, were 18 x 14 cm, which gave an effective surface of approximately 500 cm^2. For the sodium chloride solution, the pouches were 17 x 12 cm between seams which yielded an effective surface of approximately 400 cm^2.

Except for some of the early pouches, each pouch was constructed with a neck through which the test liquid could be introduced. Figure 30 shows a pattern used for cutting and seaming the pouches. The pouch seams were tested by filling the pouches with water and then letting them stand for several days. Initially, a relatively large number of pouches failed this test. To simulate approximately two years of exposure in a moist environment, the six pouches that were subsequently filled with acidic waste "HNO_3-HF-HOAc" were immersed for two weeks in water at 70°C. Deionized water was added to the CPE pouch after one week to prevent the pouch walls from sticking together. The soaking of the pouches at 70°C further tested the integrity of the seams. After a pouch was filled with a waste or test liquid, as much air as possible was removed and the neck was sealed. Great

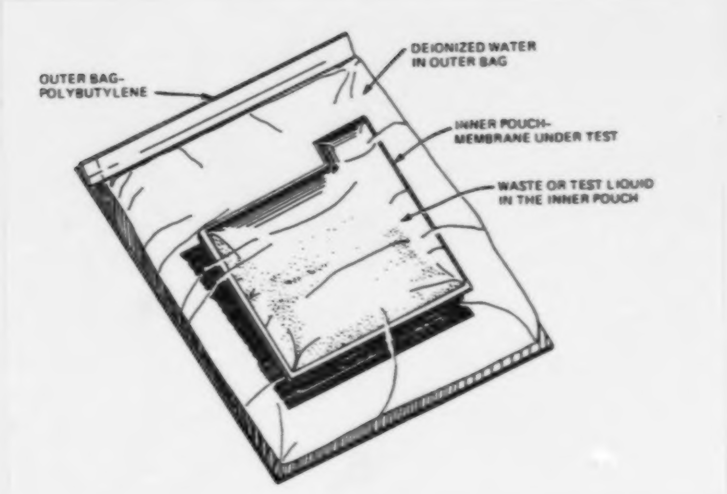

OUTER BAG-
POLYBUTYLENE
DEIONIZED WATER
IN OUTER BAG
INNER POUCH-
MEMBRANE UNDER TEST
WASTE OR TEST LIQUID
IN THE INNER POUCH

Figure 29. Schematic of a pouch assembly, showing inner pouch made of membrane material under test. The pouch is filled with waste liquid and sealed at the neck. The outer polybutylene bag, which can be easily opened, is filled with deionized water. The water in the outer bag is monitored for pH and conductivity; the pouch is monitored for weight change.

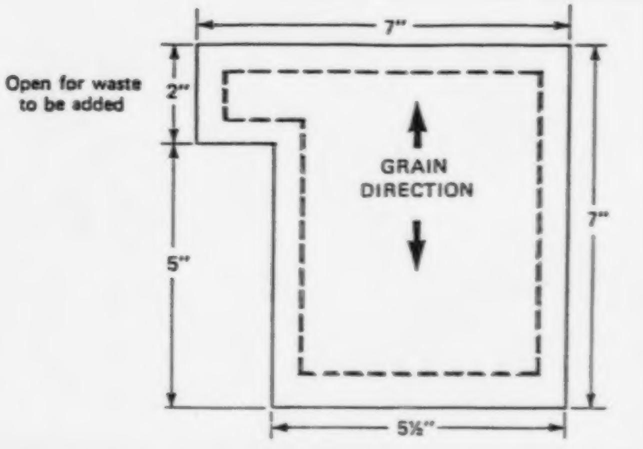

7"
Open for waste
to be added
2"
GRAIN
DIRECTION
7"
5"
5½"

Figure 30. Pattern for cutting pieces of membranes for making the pouches. Dotted line indicates the heat seal of the pouch. The inside dimensions of the pouch are 4.5 in. x 5.75 in. (11.43 x 14.6 cm) which yielded an effective area (2 sides) of ca 52 in.^{2} (335 cm^2).

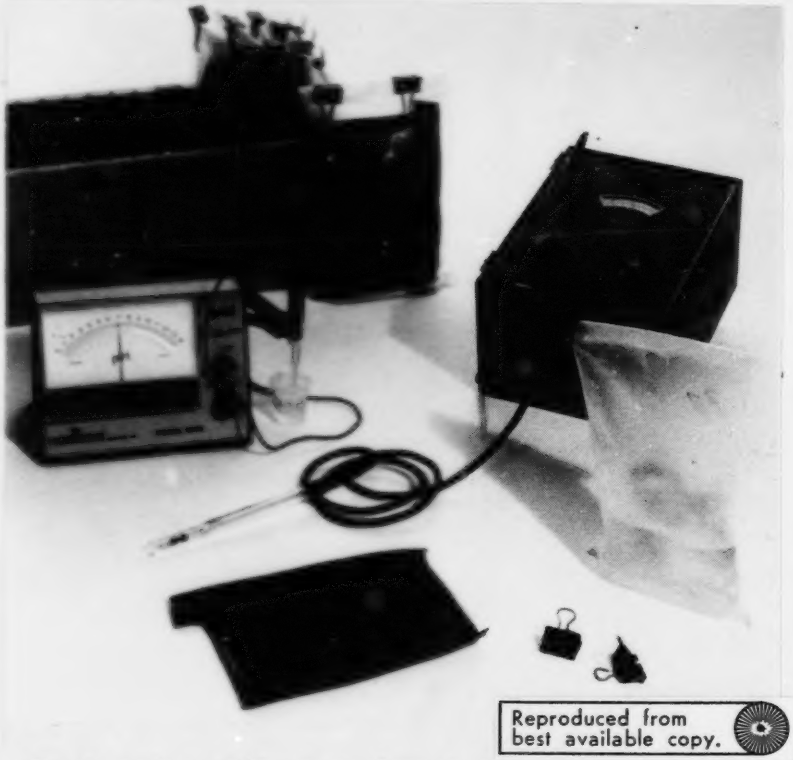
care was taken to keep the inside of the neck dry to ensure a good seal. Approximately 200 mL of waste liquid were added to the pouches. Most of the pouches were heat-sealed and the rest were sealed with either an adhesive or a solvent.

Monitoring of Pouches During Exposure

To monitor the pouches, the following tests were performed during the exposure:

1. The deionized water in each of the outer bags was tested periodically for pH, electrical conductivity, and for odor, e.g. butyric acid as in the case of tests with MSW leachate.
2. The pouches containing the test liquids were removed periodically from the water, wiped dry, inspected for possible leaks, and weighed.

Figure 31 shows the pouches, the tray assembly for holding the pouches, and the auxiliary equipment for monitoring the pouches.



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best available copy.

Figure 31. Pouch and auxiliary equipment for monitoring the pouches of polymeric membrane liners to assess permeability to water and constituents of waste liquids. Monitoring equipment for measuring pH and electrical conductivity of the water in the outer bags is shown.

During the exposure, evaporation of the water in the outer bags would cause the water level to drop exposing the top part of the pouch to air. The loss was replaced with additional deionized water. Also, if the concentration of ions in the outer bag appeared to be too high due to migration or possibly to a leak, the entire contents were replaced with deionized water.

Dismantling of Pouch After Exposure

Most of the pouches were allowed to continue their exposure for comparatively long times in order to assess the effects of exposure to water and wastes. If a leak developed in the pouch, as indicated by abrupt changes in the monitoring data, the pouch would be withdrawn from exposure, emptied, and the liner and liquids tested.

Dismantling of the pouches involved the following steps:

1. Weigh the pouch bag.
2. Determine pH and electrical conductivity of the water in the outer bag.
3. Measure length and width between seams of pouch.
4. Empty pouch and weigh contents.
5. Determine pH and electrical conductivity of waste removed from pouch.
6. Dismantle pouch at seams, leaving bottom seam intact for measurement.
7. Blot the pouch dry and weigh to determine weight increase.
8. Prepare specimens for physical tests. A pattern used for dieing out specimens in shown in Figure 32.

Tests of Exposed Membranes from Pouches

The following tests were performed on the exposed membranes:

1. Determination of percent volatiles (two hours at 105°C).
2. Determination of the percent extractables, Matrecon Test Method 2 (Matrecon, 1983, pp. 340-43).
3. Tensile in machine and transverse directions, three specimens each direction, ASTM D412.
    - Tensile strength, psi.
    - Elongation at break, percent.
    - Tensile set after break, percent.
    - Stresses at 100 and 200% elongation, psi.

4. Tear resistance in machine and transverse directions, two specimens each direction, ASTM D624, Die C.

5. Puncture resistance, two specimens, FTMS 101B, Method 2065.

6. Hardness, Duro A (Duro D if Duro A reading is greater than 80), ASTM D2240.

7. Seam strength, one specimen in peel (ASTM D1876, modified) and one specimen in shear (ASTM D882, modified).

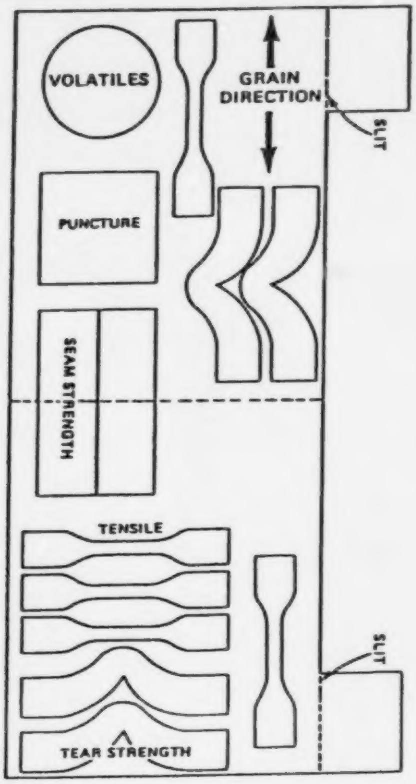
VOLATILES
GRAIN DIRECTION
SLIT
PUNCTURE
SEAM STRENGTH
TENSILE
SLIT
TEAR STRENGTH

Figure 32. A pattern used for dieing out test specimens from dismantled pouch made of a sheeting without fabric reinforcement.

Reporting of Results

The following results were reported on each pouch that was tested:

1. Properties of the unexposed membrane.

2. Properties of the membrane at the end of test, including volatiles, extractables, and physical properties.

3. Percent retention of or changes in properties during the exposure.
4. Electrical conductivity of water in outer pouch as a function of time.
5. Weight of pouch as a function of exposure time.
6. pH of water in outer bag as a function of exposure time.
7. Measurement of amount of waste liquid in pouch at time it was dismantled.
8. Analysis of waste in pouch, i.e. pH, electrical conductivity, and percent total solids.
9. Calculation of water transmission rate.

EXPERIMENTAL RESULTS

Combinations of Polymeric Membranes and Waste Liquids

Seventy-one pouches made with thermoplastic and partially crystalline membranes were tested. It was felt at the time that the feasibility and utility of the pouch test should first be thoroughly demonstrated and that later work could be initiated to develop designs and seaming methods for making pouches of crosslinked membranes and thus make it applicable to all membranes. Also, it was felt that fabric-reinforced membranes should be avoided in the initial studies.

The combinations that were put into test are shown in Table 54. They include 15 different membranes and 11 wastes. Pouches of elasticized polyolefin (136), polybutylene (98), and polyvinyl chloride (19) were filled and tested with all 11 wastes. Six different PVC membranes ranging in thickness from 11 to 33 mils were tested and include variations in manufacturers and in thickness of sheeting.

Monitoring of Pouches During Exposure

Pouches Containing Acidic Waste, "HNO_3-HF-HOAc"--

This was the first set of pouches placed in exposure under this project. These pouches contained a waste that was being used in several parts of the project. Furthermore, this set was the first to complete the entire series of tests that were planned in the basic design of the pouch test.

As indicated above, monitoring the pouch assembly included weighing the pouches and the measuring pH and electrical conductivity of the water in the bags in which the pouches were immersed. In addition, the general condition of the pouches and the waters was observed. If necessary, deionized water was added if too much water had evaporated, and the water was completely replaced if the concentration of ions and dissolved constituents appeared to have become too high.

TABLE 54. COMBINATIONS OF POLYMERIC MEMBRANES AND WASTES IN POUCH TESTS

Polymeric membrane liner		Wastes										
		Acidic		Alkaline		Brine	Industrial		Oily			Pesticide
Polymer	Number[a]	"HFL" (W-10)	"HNO_3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste" (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	"Weed killer" (W-11)
Chlorinated polyethylene	77 (30)	...	...	...	...	P6[b]	...	...	...	...	...	...
	86 (22)	P45	P19	P32	P26	P77	P71	...	...	P63	P53	...
Chlorosulfonated polyethylene	6R (31)	...	...	...	...	P2[b]	...	...	...	P56	...	...
	55 (35)	...	...	...	...	P76	P70	...	...	P62	...	...
	85 (33)	...	P18	P31	P25	...	...	...	...	...	...	...
Elasticized polyolefin	36 (22)	P44	P17	P30A,30B	P24	P4[b]	P69	P80	P85	P61	P52	P83
Polyester elastomer	75 (8)	...	...	...	...	P7[b]	...	...	...	...	...	...
Polybutylene	98 (8)	P47	P15	P28	P22	P78	P72	P81	P86	P65	P55	P84
Polyethylene Low density	21 (10)	P43	...	...	...	P75	P68	...	...	P60	P51	...
Polyvinyl chloride	11 (30)	...	...	...	...	P3[b]	...	...	...	...	...	...
	17 (20)	P48	...	...	...	P73	P66	...	...	P57,58	P49	...
	19 (22)	P42	P16	P29	P23	P74	P67	P79	P87	P59	P50	P82
	59 (33)	...	...	...	...	P5[b]	...	...	...	...	...	...
	88 (20)	...	P20	P33	P27	...	...	...	...	...	...	...
	93 (11)	P46	...	...	...	...	...	...	...	P64	P54	...

[a] Matrecon liner number and thickness in mils (in parentheses).

[b] Results reported in Haxo et al, 1983. P = pouch number.

9-9

Selected results of the monitoring are presented in Table 55 and the data on the weight increases, pH, and electrical conductivity are presented in Figures 33 through 35. In this set of 6 pouches, only one failed, i.e. the polybutylene, which failed due to cracking and embrittlement caused by exposure of the top of the pouch to light.

Note: The compartmentalized trays that contained the pouch assemblies during exposure were kept on a table in the laboratory and exposed to both sunlight and fluorescent light. All pouches are now kept under cover in closed cabinets.

The polybutylene sheeting did not contain carbon black.

These data clearly demonstrate the permeability of polymeric membranes to water, to hydrogen ions, and possibly to other constituents. Also, they show the difference in permeability of the membranes. These results are discussed more fully in the following subsections.

Monitoring of Other Pouches--

Sixty-five additional pouches were involved in the testing and were monitored during the course of the project. Selected results of the monitoring are presented in Table 56, which includes data at extended elapsed times or at the time of failure. Included are data on the weight gain by the pouches during the exposure and the pH and electrical conductivity of the water in the outer bags at the indicated elapsed time. In addition, comments are included with respect to the condition of the pouch, such as softening, stiffening, color, and the presence of oil on the outside surface. With respect to the water in the outer bag, comment is made on odor, color of the water, and the presence of suspended material or oil. The pouches that contained oily wastes generally were found to swell, soften, and exude oil on the outer surface.

Of particular interest are the results for Pouches 30A and 30B which were fabricated of elasticized polyolefin and contained the highly alkaline waste, "Slop water" (W-4). The first pouch (P30A) showed a low permeability during the first year and then a much higher permeability subsequently until it failed after approximately 790 days of exposure. The pouch increased substantially in weight and the electrical conductivity of the water in the outer bag increased to 14,000 per μmho and the pH to 12.5. A second pouch (P30B), which replaced the first and contained the same waste, has performed similarly, and had not failed after 1670 days. As in the case of the first pouch, its permeability increased from a relatively low value to a high value after about a year of exposure.

Failure of Pouches During Exposure--

In spite of the testing of the pouches before filling with wastes, 13 of the 71 pouches failed prematurely, ranging from one day to 872 days. All of the failures, except the failure of the one polybutylene pouch, were in or at the seams. Those that failed at one and nine days were replaced.

TABLE 55. POUCH TEST OF THERMOPLASTIC AND PARTIALLY CRYSTALLINE POLYMERIC MEMBRANES WITH ACIDIC WASTE, "HNO_3-HF-HOAc"

Chlorosulfonated Polyethylene, Chlorinated Polyethylene, Elasticized Polyolefin, Polybutylene, and Polyvinyl Chloride

Monitoring of Pouches

Liner and pouch	Exposure time, days																
	1	5	40	93	143	386	500	552	625	790	900	949	1160	1174	1199	1427	1887
Chlorinated polyethylene (L-86, P19)[a]																	
Change in weight, g	-0.7	-0.52	0.01	0.65	1.05	6.78	15.78	20.28	26.58	38.27	46.38	52.34	62.98	...	65.43	79.68	100.00
pH	5.9	6.0	5.9	5.8	5.6	2.7	2.0	1.6	2.2	1.8	1.8	1.8	2.1	3.1	2.6	2.0	1.8
Electrical conductivity, μmho	8.5	14.8	19.0	23.4	32.2	420	3550	5500	6300	8500	9900	12,300	14,600	350	900	4000	5900
Chlorosulfonated polyethylene (L-85, P18)																	
Change in weight, g	-1.61	-1.11	1.38	3.11	4.53	8.38	10.18	10.68	12.98	17.60	20.77	25.36	28.56	...	30.86	39.68	56.88
pH	6.6	6.8	7.1	7.5	7.6	7.0	7.8	8.4	7.8	7.3	3.0	2.6	2.8	3.3	2.8	2.3	2.0
Electrical conductivity, μmho	6.3	8.8	21.7	42.7	71	430	825	1210	1140	1800	1145	1710	2500	310	600	1860	3600
Elasticized polyolefin (L-36, P17)																	
Change in weight, g	0.01	0.01	0.07	0.22	0.19	0.59	0.79	0.69	0.89	1.18	1.33	1.42	1.37	...	1.69	2.09	3.58
pH	6.4	6.4	6.3	6.4	6.1	5.6	5.4	5.8	4.6	4.7	4.4	4.2	4.1	5.6	4.9	4.9	3.3
Electrical conductivity, μmho	4.1	5.4	8.4	9.5	11.9	26.0	230	115	60	50	69	92	92	1	8	35	80
Polybutylene (L-98, P15)																	
Change in weight, g	0.02	0.00	0.05	0.29	0.24	0.82	0.82	0.82	58.22	...	...	...	...	...	...	...	...
pH	5.5	5.3	3.8	3.4	3.2	2.6	2.8	2.6	3.0	...	...	...	...	...	...	...	...
Electrical conductivity, μmho	2.7	4.4	42.0	98	174	350	410	440	440	...	...	...	...	...	...	...	...
Polyvinyl chloride (L-19, P16)																	
Change in weight, g	-0.15	0.00	0.38	1.23	1.84	5.50	7.10	7.40	8.80	10.78	9.60	13.27	14.70	...	15.35	17.50	22.5
pH	5.5	5.6	...	3.4	2.8	2.0	2.1	1.8	2.5	2.2	2.2	2.4	2.2	3.3	2.8	2.2	2.0
Electrical conductivity, μmho	11.4	13.2	34	113	480	2000	2750	3150	2900	3290	3900	3950	4800	163	420	450	3200
Polyvinyl chloride (L-88, P20)																	
Change in weight, g	0.02	0.26	1.11	3.00	5.43	12.93	15.73	17.23	19.43	23.08	26.16	28.47	31.07	...	32.21	37.03	45.43
pH	4.9	4.9	3.3	2.8	2.7	1.8	2.0	1.7	2.3	...	2.0	1.9	2.5	3.1	2.7	2.1	1.9
Electrical conductivity, μmho	18.6	24.0	153	330	647	3200	4500	4900	4700	4800	5400	6700	9000	361	680	2570	4600

[a] L = liner number; P = pouch number.

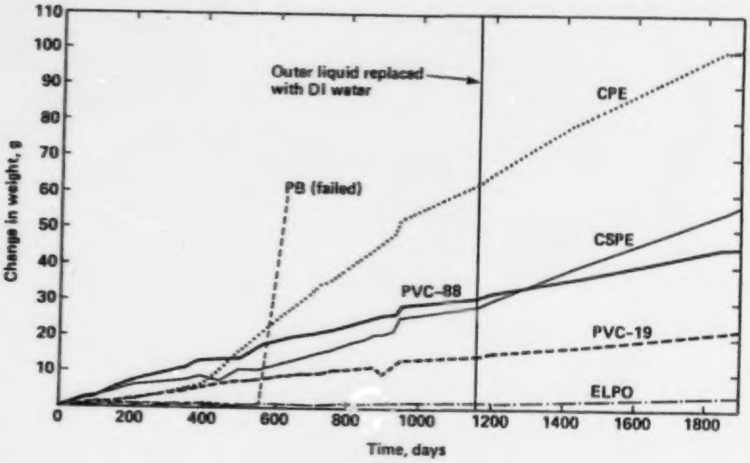

Figure 33. Monitoring of pouches that contained the highly acidic waste, "HNO_3-HF-HOAc" (W-9). Weight changes of the individual pouches P15 - P20 plotted against exposure time in days.

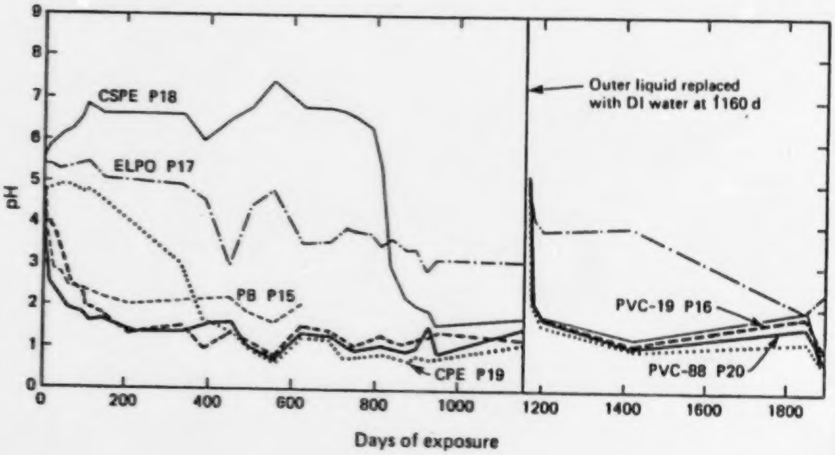

Figure 34. Monitoring of pouches that contained the highly acidic waste, "HNO_3-HF-HOAc" (W-9). pH of the deionized water in the outer polybutylene bags in which pouches P15 - P20 were immersed is plotted against exposure time in days.

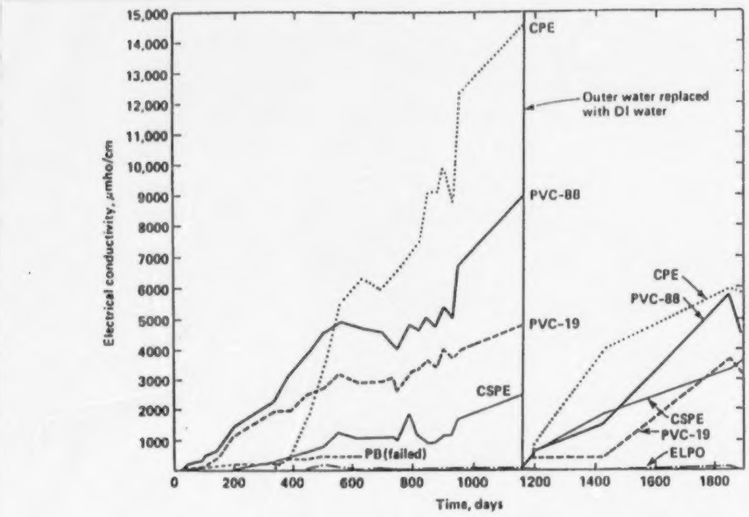

Figure 35. Monitoring of pouches that contained the highly acidic waste, "HNO_3-HF-HOAc" (W-9). Electrical conductivity of the deionized water in the outer polyethylene bags containing pouches P15 - P20 is plotted against exposure time in days.

Also replaced after approximately 790 days was P30A which was made of elasticized polyolefin and was filled with the highly alkaline waste "Slop water" (W-4).

The incidence of seam failure appears to be predominantly among those made of thick membranes, those seamed with solvents, and those that contained "Slop water" waste. There were no seam failures among the heat-sealed polybutylene and polyethylene pouches. Seam failures are indicative of problems in the making of pouches; they do not reflect on the seaming by manufacturers of the liners, either in the factory or in the field. Since all of the heat-sealed pouches that failed were fabricated with a device that was not intended for seaming such thick materials, these failures can only be a reflection of the problem of using improper equipment.

Subsequently, we obtained a heat-sealing device with which we have been more successful in preparing the seams of the thicker materials. This latter device is also primarily in tended for making polyethylene bags; it is semiautomated and can be run at higher temperatures under controlled pressures and times.

Note: The above heat-sealing unit is the "PAC Model 12 PI Impulse Bag Sealer" purchased from Packaging Aids Corporation, San Francisco, California 94107.

TABLE 56. MONITORING OF POUCHES

pH and Electrical Conductivity of Water in Outer Bag and Weight Changes of Filled Pouches

Waste	Polymer	Liner number[a]	Pouch number	Elapsed time, d	pH	Electrical conductivity, μmho	Change in weight	Comments
Acidic - "HFL" (W-10)	Chlorinated polyethylene	86 (22)	P45	1833	4.1	510	11.3	Pouch wrinkled, coated w/brown green slime.
	Elasticized polyolefin	36 (22)	P44	1833	3.2	60	0.4	...
	Polybutylene	98 (8)	P47	1833	2.9	155	0.7	...
	Polyethylene - low density	21 (10)	P43	1833	2.7	580	1.2	...
	Polyvinyl chloride	17 (20)	P48	1823	2.5	540	4.4	...
	Polyvinyl chloride	19 (22)	P42	1833	2.6	280	3.9	Pouch wrinkled.
	Polyvinyl chloride	93 (11)	P46	1833	2.8	595	9.8	Pouch discolored, swollen.
Alkaline - "Slop water" (W-4)	Chlorinated polyethylene	86 (22)	P32	65[b]	7.9	270	2.7	Leak noted at 93 d. Dismantled at 625 d.
	Chlorosulfonated polyethylene	85 (33)	P31	2044	9.3	1800	36.5	Pouch wrinkled; blisters; odor in water.
	Elasticized polyolefin	36 (22)	P30A	753[b]	12.4	10,800	213.6	Leaked at seam at 790 d; blisters noted.
	Elasticized polyolefin	36 (22)	P30B	1350	11.1	5460	227.8	Pouch swollen; has yellow crystals on it.
	Polybutylene	98 (8)	P28	2044	9.4	1950	16.8	Pouch has small blisters.
	Polyvinyl chloride	19 (22)	P29	872[b]	12.5	14,700	152.4	Pouch very stiff; seam failed at 930 d.
	Polyvinyl chloride	88 (20)	P33	65[b]	9.6	235	...	Leak noted at 93 d, dismantled at 625 d.
Alkaline - "Spent caustic" (W-2)	Chlorinated polyethylene	86 (22)	P26	2044	3.7	102	22.4	Pouch wrinkled; coated w/brown green slime.
	Chlorosulfonated polyethylene	85 (33)	P25	2044	4.2	23	22.5	Pouch wrinkled.
	Elasticized polyolefin	36 (22)	P24	2044	4.1	73	4.2	...
	Polybutylene	98 (8)	P22	2044	4.1	16	12.6	...
	Polyvinyl chloride	19 (22)	P23	2044	4.8	660	60.3	...
	Polyvinyl chloride	88 (20)	P27	2044	3.3	94	54.9	Pouch seams wrinkled, swollen
Brine - 5% NaCl (W-19)	Chlorinated polyethylene	77 (30)	P6	...	...	...	...	...
	Chlorinated polyethylene	86 (22)	P77	316	3.6	86	4.0	Wrinkled at seams.
	Chlorosulfonated polyethylene	6R (31)	P2	1151	7.4	585	10.2	Swollen.
	Chlorosulfonated polyethylene	55 (35)	P76	315	6.7	66	4.7	...
	Elasticized polyolefin	36 (22)	P4	1151	4.8	34	2.3	...
	Polybutylene	98 (8)	P78	316	5.1	27	0.7	Wrinkled.
	Polyester	75 (8)	P7	928[b]	4.5	71	25.9	Leak noted at 955 d.
	Polyethylene - low density	21 (10)	P75	315	4.4	31	0.9	...
	Polyvinyl chloride	11 (30)	P3	338[b]	...	22	0.4	Leak in seam at thin spot[c] noted at 791 d.
	Polyvinyl chloride	17 (20)	P73	310	3.1	371	6.5	...
	Polyvinyl chloride	19 (22)	P74	315	6.4	16	1.8	...
	Polyvinyl chloride	59 (33)	P5	1152	5.4	4500	2.1	...

Continued

TABLE 56. Continued

Waste	Polymer	Liner number[a]	Pouch number	Elapsed time, d	pH	Electrical conductivity, µmho	Change in weight	Comments
Industrial - "Basin F" (W-16)	Chlorinated polyethylene	86 (22)	P71	38[b]	4.3	38	1.0	Outer bag failed at 49 d.
	Chlorosulfonated polyethylene	55 (35)	P70	1641	8.3	3600	10.8	Pouch wrinkled at edges.
	Elasticized polyolefin	36 (22)	P69	1641	7.3	189	2.8	...
	Polybutylene	98 (8)	P72	1641	7.7	230	4.8	...
	Polyethylene - low density	21 (10)	P68	1641	6.0	330	10.9	...
	Polyvinyl chloride	17 (20)	P66	227[b]	4.7	33	7.5	Pouch smooth; leak in seam at 727 d.
	Polyvinyl chloride	19 (22)	P67	1641	8.0	600	29.7	Pouch slightly wrinkled.
"Lead waste" (W-14)	Elasticized polyolefin	36 (71)	P80	277[b]	5.2	11	-0.9	Pouch smooth; leak in seam at 726 d.
	Polybutylene	98 (8)	P81	1638	7.9	315	-1.4	...
	Polyvinyl chloride	19 (22)	P79	1638	4.7	46	-1.2	...
Oily - "Slurry oil" (W-15)	Elasticized polyolefin	36 (22)	P85	1638	3.2	59	0.0	Pouch wrinkled; oil on surface; stained.
	Polybutylene	98 (8)	P86	1638	3.7	56	0.1	Pouch sticky; oil on surface; stained.
	Polyvinyl chloride	19 (22)	P87	1638	4.0	42	0.6	Pouch wrinkled; oil on surface[d].
Oily - "Oil Pond 104" (W-5)	Chlorinated polyethylene	86 (22)	P63	1485	4.5	190	1.81	Pouch flat.
	Chlorosulfonated polyethylene	6R (31)	P56	1494	5.5	310	16.3	Pouch swollen, blistered.
	Chlorosulfonated polyethylene	55 (35)	P62	1485	5.5	320	12.9	Pouch flat, puckered.
	Elasticized polyolefin	36 (22)	P61	1485	3.2	140	-1.3	Pouch flat, possible seam leak.
	Polybutylene	98 (8)	P65	1485	3.4	130	-4.5	Oil on pouch outer surface.
	Polyethylene - low density	21 (10)	P60	1494	3.2	130	-4.3	Oil on pouch outer surface.
	Polyvinyl chloride	17 (20)	P57	1	...	...	...	Failed at 1 d.
	Polyvinyl chloride	17 (20)	P58	1485	2.8	330	-0.3	...
	Polyvinyl chloride	19 (22)	P59	1485	3.8	49	-1.2	Pouch puckered and stiff.
	Polyvinyl chloride	93 (11)	P64	1[b]	5.9	6	0.0	Seam leaked at 9 d.
Oily - "Weed oil" (W-7)	Chlorinated polyethylene	86 (22)	P53	1654	4.0	420	10.3	...
	Elasticized polyolefin	36 (22)	P52	1654	3.3	52	0.3	...
	Polybutylene	98 (8)	P55	1654	4.4	39	-0.5	Brown sediment in pouch bottom.
	Polyethylene - low density	21 (10)	P51	1654	3.4	46	0.9	Brown sediment in pouch bottom.
	Polyvinyl chloride	17 (20)	P49	1640	2.5	448	0.3	Pouch has small blisters.
	Polyvinyl chloride	19 (22)	P50	1654	4.8	56	-0.2	Pouch has small blisters.
	Polyvinyl chloride	93 (11)	P54	62[b]	3.8	54	2.2	Seam open; pouch flat; noted at 78 d.
Pesticide - "Weed killer" (W-7)	Chlorinated polyethylene	36 (22)	P83	1638	3.2	67	0.3	...
	Elasticized polyolefin	98 (8)	P84	1638	3.3	46	0.1	...
	Polyvinyl chloride	17 (20)	P82	1638	3.4	42	-0.2	White rubbery sediment in pouch.

[a] Matrecon liner serial number. Thickness, in mils, in parentheses.
[b] Leak detected in bag during next monitoring.
[c] Thinness probably related to overheating during heat-sealing.
[d] This pouch was viewed under ultraviolet light and the oil flouresced.

Other Observations--

Many of the pouches that contained oily wastes appeared to have been permeated by the oily constituents. The outsides of the pouches were tacky and oily and, in some cases, the water in the outer bag had a strong oily odor. It is apparent that some of the organic wastes have permeated the pouch walls and dissolved in the water in the outer bags. However, direct analyses of these waters were not made.

Dissecting of Pouches Containing the "HNO_3-HF-HOAc" Acidic Waste

Condition of the Pouch Assemblies--

As indicated above, the pouch (P15) made of polybutylene failed at 609 days and was removed and tested at 625 days. Five of the original pouches survived to 1887 days of exposure. These were dissected so that the pouches and the contents of the pouches could be weighed, the contents analyzed, and the properties of the pouch walls measured.

All of the pouches either had retained approximately their original flexibility or had swollen and softened during the exposure period. The water in the outer bags had a chemical or acrid odor indicating migration of constituents from the waste liquid in the pouches to water in the outer bag.

The CPE pouch (P19) appeared to be in good shape at the time of dismantling, although the seams were relatively easy to pull apart. The surface on the inside of the pouch was somewhat pitted. The membrane had obviously swollen.

The CSPE pouch (P18) had blistered badly on one side. This sheeting was not fabric-reinforced. The liquid contents of the blisters were removed with a hypodermic needle and analyzed for pH and electrical conductivity. The pH of the liquid was 1.35 and the electrical conductivity of a 1:1 dilution with deionized water was 12,400 µmho. The waste fluid obviously permeated into the sheeting which delaminated relatively easily in the area of the blisters when pulled by hand.

The pouch made of elasticized polyolefin (P17) retained its original condition well during exposure. It appeared to have changed little during the 1887 days of exposure.

The pouch made of polybutylene (P15), which had failed at 609 days and was dismantled at 625 days, was badly cracked at the upper seam due to light degradation. The portion of the pouch that was below the water level of the outer bag appeared to have retained its flexibility and original properties.

Pouch 16, made of PVC liner No. 19, was flexible but the inside was sticky. Also, the seams which, had been made with a solvent seal of 50:50 tetrahydrofuran:trichloroethane, were easy to peel apart.

Pouch 20, made of PVC liner No. 88, had stiffened somewhat during exposure. The outside surface had become rough and discolored compared with the unexposed material. The inside was considerably more discolored and pitted, indicating some interaction between the membrane and the waste liquid.

Weights--

The various weights of the pouches and the contents of the pouches are presented in Table 57. These data confirm the monitoring data; they show substantial increases in the contents of the pouches of CPE (P19), CSPE (P18), PVC (P16), and PVC (P20), further demonstrating the movement of water from the outer bag into the pouch due to the concentration differences between the liquids on the two sides of the membrane.

The pH and electrical conductivity measurements of the water in the outer bags also show the movement of the hydrogen ions out of the pouches into the water in which the pouches were immersed. The lower conductivity and higher pH of the waste removed from the pouches at the end of the exposure period when compared with the waste placed in the pouches at the beginning further confirm this. The elasticized polyolefin pouch wall had a particularly low permeability to water and the dissolved consitituents of the waste.

Analysis of the Contents of the Pouches and of the Outer Bags--

On opening the pouches the contents were collected, weighed, and tested for pH and electrical conductivity. The results, presented in Table 57, show a substantial increase in the volume and a significant decrease in the electrical conductivity of the contents, indicating a dilution by the water that had entered through the pouch walls. Calculations of the transmission of the water through the pouch walls were made and are reported in Table 57. The calculations show the chlorinated polyethylene to be the most permeable of the group and the elasticized polyolefin the least.

These transmission values correlate well with the values calculated from the weight increases of the pouches during the exposure. However, the fact that part of the increases in weight are a result of the absorption of water by the pouch walls requires that the earlier values be corrected. Although the polybutylene bag failed prematurely, the transmission value based on the weight gains were low.

Physical Properties of the Pouch Walls--

After their contents were removed, the pouches were cut, washed, and tested as described in the basic pouch test procedure. These tests included analytical properties, i.e. volatiles and extractables, and physical properties, i.e. tensile, tear, puncture strength, hardness, and seam strength. Results of these tests are presented in Table 58, and the percent retentions of properties are presented in Table 59.

TABLE 57. POUCH TEST OF THERMOPLASTIC AND PARTIALLY CRYSTALLINE POLYMERIC MEMBRANES WITH ACIDIC WASTE, "HNO_3-HF-HOAc" (W-9)

Chlorosulfonated Polyethylene, Chlorinated Polyethylene, Elasticized Polyolefin, Polybutylene, and Polyvinyl Chloride Data on Pouch and Waste Before and After Dismantling

Polymer	Chlorinated polyethylene	Chlorosulfonated polyethylene	Elasticized polyolefin	Polybutylene	Polyvinyl chloride	
Matrecon liner serial number	86	85	36	98	19	88
Liner thickness, mil	22	33	22	8	22	20
Pouch number	P19	P18	P17	P15	P16	P20
Number of days exposed	1887	1887	1887	609[a]	1887	1887
Effective area of pouch (seam-to-seam) of exposed pouch, m^2	0.0431	0.0484	0.0446	...	0.0443	0.0523
Weight of empty pouch						
Unexposed, g	39.17	59.03	27.32	8.24	33.24	31.68
Conditioned[b], g	57.44	79.46	26.70	8.82	34.09	33.45
After exposure, g	53.45	87.49	28.35	...	35.18	43.14
Change from unexposed, g	14.28	28.46	1.03	...	1.94	11.46
Change from conditioned, g	-3.99	8.03	1.65	...	1.09	9.69
Change from unexposed, %	36.5	51.8	3.8	...	5.8	36.2
Weight of filled pouch						
At beginning of exposure, g	250.22	261.93	128.31	122.98	176.60	189.47
After exposure, g	350.22	318.80	131.89	...	199.10	234.90
Change in weight, g	100.00	58.87	3.58	...	22.50	45.43
Waste contents of pouch						
Weight at beginning of exposure, g	192.79	182.47	101.61	114.16	142.51	156.02
Weight after exposure, g	291.49	229.04	103.01	...	163.54	191.86
Change in weight, g	98.70	46.57	1.40	...	21.03	35.84
pH after exposure[c]	1.10	1.00	0.95	...	0.90	1.05
Electrical conductivity[d], after exposure, µmho	110,000	170,000	170,000	...	170,000	140,000
Liquid in outer bag						
Volume at conclusion of test, mL	375	395	520	...	463	400
pH at conclusionn of test	1.80	1.95	3.25	3.0	2.0	1.85
Electrical conductivity						
Before water change[f], µmho	14,600	2,500	92	...	4,800	9,000
At conclusion of test, µmho	5,900	3,600	86	440	3,200	4,600
Water transmission through pouch wall, $g/m^2/day$	1.213	0.510	0.017	...	0.252	0.363

[a] Pouch failed due to cracking and embrittlement at upper seam caused by light degradation. Film was unpigmented, i.e. it contained no carbon black to absorb ultraviolet light.

[b] Empty pouches conditioned for two weeks in water at 70°C before waste added. Water added to CPE pouch (P19) after one week to prevent sides from sticking together.

[c] pH at beginning of test = 0.9.

[d] Electrical conductivity of waste at beginning of test = 200,000 µmho.

[e] The volume of liquid in outer bag varied in course of exposure due to evaporation. Deionized water was added periodically to cover the pouches.

[f] Outer liquid replaced with deionized water after 1169 days of exposure.

TABLE 58. POUCH TEST OF THERMOPLASTIC AND PARTIALLY CRYSTALLINE POLYMERIC MEMBRANES WITH ACIDIC WASTE, "HNO_3-HF-HOAc" (W-9)

Chlorosulfonated Polyethylene, Chlorinated Polyethylene, Elasticized Polyolefin, Polybutylene, and Polyvinyl Chloride
Properties of Membranes after Exposure

Polymer	Direction of test	Chlorinated polyethylene	Chlorosulfonated polyethylene	Elasticized polyolefin	Polybutylene	Polyvinyl chloride	
Matrecon liner serial number		86	85	36	98	19	88
Nominal thickness, mil		22	31	22	8	22	20
Exposure time, days		1887	1887	1884	609	1887	1887
Analytical properties							
Volatiles , %		46.53	34.39	8.26	...	12.33	30.35
Extractables, %		...	...	7.73	4.70	38.58	34.12
Physical properties							
Average thickness, mil		35.9	41.6	23.1	10	22.6	26.1
Tensile at break, psi	Machine	760	860	2455	5610	2290	1865
	Transverse	460	935	1985	5690	2120	1600
Elongation at break, %	Machine	265	180	680	350	345	240
	Transverse	315	185	620	390	355	250
Set after break, %	Machine	85	20	455	200	80	50
	Transverse	45	30	375	235	90	45
S-100, psi	Machine	405	425	895	2830	1285	1255
	Transverse	160	470	845	2790	1230	1130
S-200, psi	Machine	625	...	975	3770	1705	1690
	Transverse	270	...	925	3420	1580	1470
Tear strength, ppi	Machine	85	...	380	...	290	265
	Transverse	60	80	350	...	295	210
Puncture strength							
Thickness, mil		39.0	40.0	24.4	...	23.8	26.5
Stress, lb		11.4	42.3	36.4	...	38.4	20.4
Elongation, in.		0.70	0.73	1.22	...	0.84	0.40
Hardness (durometer), 5-second reading		45A	54A	85A	...	72A	74A
		...	...	28D	...	...	...
Seam strength							
Bonding system		Heat	Adhesive	Heat	Heat	Solvent[a]	Heat
In shear, ppi		11.7	...	...	...	28.0	25.8
Locus of failure[b]		LS	...	...	...	SE	SE
In peel, ppi		2.2	...	20.6	...	6.0	11.6
Locus of failure[b]		LS	...	SE	...	LS	LS

[a] 50:50 tetrahydrofuran:trichloroethane mixture.

[b] LS = Failed at the bond between the adhered liner surfaces. SE = failed at the seam edge.

TABLE 59. POUCH TEST OF THERMOPLASTIC AND PARTIALLY CRYSTALLINE POLYMERIC MEMBRANES WITH ACIDIC WASTE, "HNO_3-HF-HOAc" (W-9)
Chlorosulfonated Polyethylene, Chlorinated Polyethylene, Elasticized Polyolefin, Polybutylene, and Polyvinyl Chloride
Retention of Original Values

Polymer	Direction of test	Chlorinated polyethylene	Chlorosulfonated polyethylene	Elasticized polyolefin	Polybutylene	Polyvinyl chloride	
Liner number		86	85	36	98	19	88
Nominal thickness, mil		22	31	22	8	22	20
Exposure time, days		1887	1887	1884	609	1887	1887
Analytical properties							
Volatiles							
Unexposed, %		0.05	0.92	0.15	...	0.05	0.17
Exposed, %		46.53	34.39	8.26	...	12.33	30.35
Extractables							
Unexposed, %		6.02	8.22	5.50	4.42	38.91	33.46
Exposed, %		...	...	7.73	4.70	38.58	34.12
Physical properties							
Thickness, % of original		167	126	100	...	108	131
Tensile at break, % ret.	Machine	41	37	93		82	55
	Transverse	30	45	78		94	55
Elongation after break, % ret.	Machine	75	69	101	...	105	74
	Transverse	53	57	95	...	104	75
Set after break, % ret.	Machine	41	12	99	...	82	49
	Transverse	19	16	87	...	86	45
S-100, % ret.	Machine	47	37	102	...	112	67
	Transverse	58	63	98	...	116	71
S-200, % ret.	Machine	52	...	100	...	93	65
	Transverse	67	...	96	...	99	67
Tear strength, % ret.	Machine	45	...	98	...	98	57
	Transverse	34	29	95	...	107	45
Puncture strength							
Stress, % ret.		55	88	138	...	160	71
Elongation, % ret.		0.70	0.73	1.22	...	0.84	0.40
Hardness (durometer), change in 5-second reading		-22A	-25A	+2A	...	0A	-6A

Comparison Between the Pouch Test and the Immersion Test--

The increases in weight of the pouch walls relate to the weight gains obtained in immersion-type tests. However, there may be significant differences as shown in Table 60 which compares the weight increases of the pouch walls with the weight increases of the same or similar membranes in immersion tests. Most of the weight gains in the pouch test were higher than those in the immersion tests with acidic waste, "HNO_3-HF-HOAc" and deionied water. However, this may reflect: (1) the longer exposure time, 1887 days vs 751 days, and (2) the absorption of deionized water which is generally greater than that of an aqueous solution containing dissolved salts.

TABLE 60. WEIGHT CHANGE OF MEMBRANES DURING EXPOSURE

Comparison of Pouch and Immersion Tests

Liner		Pouch test[a]		Immersion test		
Polymer	Number[b]	Time, days	Weight change, %	Time days	Weight change in waste[c], %	Weight Change in deionized water, %
Chlorinated polyethylene	77 (30)	...	...	751	19.9	12.4
	86 (22)	1887	36.4	...	...	...
Chlorosulfonated polyethylene	55 (35)	...	...	751	10.9	18.9
	85 (33)	1887	48.2	...	...	...
Elasticized polyolefin	36 (22)	1887	3.8	751	7.57	0.6
Polyvinyl chloride	11 (33)	...	...	751	22.1	-1.6
	19 (22)	1887	5.8	...	...	...
	59 (33)	...	...	751	-6.12	-0.5
	88 (20)	1887	36.2	751	28.2	-0.1

[a] Pouches contained acidic waste "HNO_3-HF-HOAc" and were immersed in deionized water. Pouches included seams.

[b] Matrecon liner number and thickness, in mils, in parentheses.

[c] Acidic waste "HNO_3-HF-HOAc."

In Table 61, the extractables of the pouch walls are compared with the extractables of the same or similar respective membranes in the immersion test with the acidic waste, "HNO_3-HF-HOAc." In both cases, the changes during exposure were small, except in the case of PVC liner (59) which decreased in extractables significantly during exposure (23.25% vs original of 35.87%).

TABLE 61. COMPARISON OF EXTRACTABLES OF EXPOSED MEMBRANES AFTER POUCH AND IMMERSION TESTS WITH ACIDIC WASTE, "HNO_3-HF-HOAc" (W-9)

Liner			Pouch test		Immersion test	
Polymer	Number[a]	Original value, %	Exposure time, d	Extract-ables, %	Exposure time, d	Extract-ables, %
Chlorinated polyethylene	77 (30)	9.13	...	...	751	12.04
	86 (22)	6.02	1887	...	...	...
Chlorosulfonated polyethylene	55 (35)	4.08	...	...	751	3.96
	85 (33)	...	1887	10.34	...	...
Elasticized polyolefin	36 (22)	5.50	1887	...	751	6.22
Polyvinyl chloride	11 (33)	33.90	...	...	751	32.25
	19 (22)	38.91	1887	33.46	...	...
	59 (33)	35.86	...	...	751	23.25
	88 (20)	34.46	1887	34.12	751	33.20

[a] Matrecon liner number and thickness, in mils, in parentheses.

In Table 62 the results on stress at 100% elongation (S-100) of the membranes after exposure in the pouch test are compared with those on the same or similar membranes from the immersion test with the acidic waste, "HNO_3-HF-HOAc." The results of this comparison are not clear because of the variation in the response of the individual liners to the exposure. However, in the two cases where the same sheeting was used, the values were comparable, even though the time periods were greatly different.

Permeability

The primary purpose of this test was to assess the permeability of the liner materials in a condition that simulates some of the conditions in a pond. The results indicate the extent of migration of several waste constituents simultaneously, i.e. water, dissolved ions, both cations and anions, and organics.

Water--

The clearest results in the pouch test with the acidic waste, "HNO_3-HF-HOAc," were those regarding the transmission of water. Deionized water in the outer bag was transmitted into the concentrated waste inside the pouch by osmosis resulting from the large concentration gradient across the membrane. Such results indicate that a pond lined with a membrane liner placed in a moist, perhaps saturated, environment in which the water is relatively pure would receive water from the environment outside the pond. This would be true even if there were no holes or breaks in the liner

TABLE 62. COMPARISON OF THE RETENTION OF STRESS AT 100 PERCENT ELONGATION OF MEMBRANES EXPOSED TO ACIDIC WASTE "HNO_3-HF-HOAc" IN POUCH AND IMMERSION TESTS

Liner		Original value,	Pouch test		Immersion test	
Polymer	Number[a]	psi	Exposure time, d	Retention, %	Exposure time, d	Retention, %
Chlorinated polyethylene	77 (30)	900	...	...	751	129
	86 (22)	575	1887	53	...	...
	100 (36)	617	...	...	751	45
Chlorosulfonated polyethylene	55 (35)	880	...	...	751	71
	85 (33)	950	1887	50	...	...
Elasticized polyolefin	36 (22)	923	1887	100	751	93
Polyvinyl chloride	11 (33)	1420	...	...	751	93
	19 (22)	1330	1887	114	...	...
	59 (33)	995	...	...	751	252
	88 (20)	1735	1887	69	751	70

[a] Matrecon liner number and thickness, in mils, in parentheses.

itself. However, water is not a polluting constituent. In the diffusion process, it migrates through the liner as an independent molecular species.

Permeability of Ions--

In the case of pouches and ponds, concentrated solutions are on one side and the purer water with fewer dissolved constituents is on the other. In such an environment, the concentration difference results in a gradient or driving force which causes the dissolved constituents to move from the concentrated solutions into the less concentrated. Thus, there can be a movement of ions, such as hydrogen ions, hydroxyl ions, and other cations and anions, out of the pouch or the pond into the ground. This occurs in the case of the pouches; in the tests with the highly acidic waste "HNO_3-HF-HOAc," the hydrogen ions in the pouch migrated through the walls and the pH of the outer solution dropped due to increased hydrogen ion concentration, and the electrical conductivity increased. The difference between the permeabilities of the different sheetings is quite apparent.

Organics--

Several of the wastes were oily, resulting in a film of oil being formed on the outside of the pouches and, since the oils themselves were not soluble in water, they tended to remain on the surface of the pouch and stop further movement of oils through the wall. If the oils had been soluble in the water, the concentration of the oil on the downstream side of the membrane would have been lower and migration of the oil would have continued.

DISCUSSION

Comparison of Permeability Results

In the pouch test with the acidic waste and the six different polymeric membrane liners, relative permeability data were obtained on two constituents, i.e. water and dissolved ions. These are reflected by the weight increases of the pouches, the increases in electrical conductivity, and the changes in pH during exposure. These results were presented in Figures 33 through 35.

In Table 63 the relative ordering of the permeabilities among the six polymeric membranes is presented for water and hydrogen ions as measured by pH and electrical conductivity. In the same table the relative permeabilities as measured by moisture vapor transmission in the E-96 test are also presented.

The pouch test results indicate that the elasticized polyolefin has the lowest permeability to water and to the hydrogen ions and the polybutylene the second lowest. The highest permeabilities were shown by the CPE and the two PVC's. The pouch of CPE showed a low permeability during the early stages, comparable to the values indicated by the moisture vapor transmission; however, after a year of exposure, during which the changes in weight of the pouch, the electrical conductivity, and pH were small, significant increases in weight and conductivity took place. These effects would indicate that the permeability of the CPE increased, probably due to high water absorption. It is also possible that pinholes may have developed in the membrane during the exposure period as the inside of the pouch had a pitted appearance. There was no apparent failure at the seams that would have caused a very abrupt increase in conductivity such as was encountered with the polybutylene.

There was also an indication that the relative order of permeability is not the same for all of the constitutents of a waste. In addition, it would appear that maintaining a membrane in a moist condition is necessary for assessing the long-term permeability of membranes which may slowly become affected by the waste liquid. The moisture vapor transmission test maintains one side of a membrane quite dry at 50% humidity, whereas the other side is maintained at 100%. Even under these conditions, some increases in moisture vapor transmission have been observed in long-term tests.

TABLE 63. RELATIVE ORDER[a] OF PERMEATION OF POLYMERIC MEMBRANES TO WATER AND HYDROGEN IONS, IN POUCH TEST WITH ACIDIC WASTE, "HNO_3-HF-HOAc" (W-9), AND TO MOISTURE VAPOR IN ASTM E96 TEST

Liner		Pouch status at 1887 days			Moisture vapor transmission, E96	
Polymer	Number[b]	Water[c]	Electrical conductivity[d]	pH[e]	g/m2 d	Order
Chlorinated polyethylene	86 (22)	5	6	5	0.643	3
Chloro-sulfonated polyethylene	85 (33)	4	3	2	0.438	2
Elasticized polyolefin	36 (22)	1	1	1	0.142	1
Polybutylene	98 (8)	1[f]	2[f]	...	...	...
Polyvinyl chloride	19 (22)	2	4	4	2.78	4
	88 (20)	3	5	3	2.94	5

[a] Order of increasing permeation through polymeric membranes in test. 1 = Least permeable and 6 = most permeable.
[b] Matrecon liner number and thickness, in mils, in parentheses.
[c] Based on change in weight of filled pouch for 400 d exposure and change in weight of contents of pouch for 1800 d exposure.
[d] Electrical conductivity of water in outer bags.
[e] pH of water in outer bags.
[f] Extrapolated.

Comparison of Retention of Physical Properties in Pouch and Immersion Tests

The retention of properties of the liners in the pouch and immersion tests appear to be comparable as shown in Table 62. Addition information of this type will become available when the remaining pouches are removed from exposure, measured, dissected, and tested.

Expansion of Pouch Test Method to all Polymeric Membranes

The pouch method for assessing the permeability of membranes over long periods of time and of the durability of these materials in contact with wastes appears to be a highly feasible method of assessing the membranes, though extended exposures may be required. This conclusion is based on the results with the more flexible sheetings of thermoplastic and partially crystalline materials. An expansion of this test method to crosslinked polymers and elastomers, to thicker and more rigid sheetings, and to fabric-reinforced materials appears to be desirable. To achieve good seams in the pouches may require the development of special adhesives and possibly new pouch designs which can accommodate the more rigid materials, e.g. 100 mil HDPE. It is desirable, of course, to use the commercial sheetings to fabricate these pouches and not to depend upon a molded container.

SECTION 10

PYROLYSIS ANALYSIS OF POLYMERIC MEMBRANE LINERS

As has been pointed out in Section 5, the composition of liners, even those based on a given polymer, varies from manufacturer to manufacturer. Differences can be used to identify or fingerprint a compound. Pyrolysis is a method by which such an analysis can be made; it is the process by which materials break down and lose mass on exposure to heat. This technique is useful in the determination of the composition of polymeric compounds because individual components of a mixture may be pyrolyzed selectively by proper control of the experimental conditions.

In the pyrolysis procedure, the specimen is weighed at specified points throughout the experiment. The most sophisticated mode of pyrolysis in current use is thermogravimetric analysis (TGA). This analysis provides a continuous record of the sample weight throughout the procedure and is ideally suited to applications where the kinetics of degradation are important. The use of TGA for the analysis of polymeric membrane liners has been described and test results presented (Haxo, 1983).

Membrane liners are generally compounded from four classes of materials:

1. Organic polymers, e.g. polyethylene, polyvinyl chloride.
2. Monomeric organic chemicals, e.g. plasticizers, antioxidants.
3. Inorganic fillers, e.g. clays, silicates, silica, and talc.
4. Carbon black.

The weight percent of each of these components in a given membrane liner can be accurately determined by the combination of pyrolysis with solvent extraction.

Pyrolysis under a nitrogen atmosphere will result in the loss of the monomeric organic additives and of a known proportion (experimentally determined) of the organic polymers. The carbon black and filler will not be affected. Further pyrolysis under air or oxygen will result in the loss of the carbon black and any carbonaceous residue remaining from the polymer. The filler will still generally be unaffected. Solvent extraction (Appendix M) of another specimen of the membrane gives a specific determination of the monomeric organic content, e.g. plasticizer or oil, and allows a full determination of the liner composition. Table 64 outlines the weight losses occurring under each experimental condition.

TABLE 64. LINER WEIGHT LOSSES OCCURING ON PYROLYSIS AND EXTRACTION

	Degree of loss through pyrolysis		Solvent
	Under N_2	Under O_2	extraction
Organic polymers	Partial to complete	Complete	None
Monomeric organic additives	Complete	Complete	Complete
Carbon black	None	Complete	None
Inorganic filler	None	None	None

As indicated in the preceding paragraph and in Table 64, N_2 pyrolysis of a liner may result in only partial loss of the polymeric component. This is especially true of polymers containing chlorine, other halogens, or nitrogen. These materials will not smoothly depolymerize and volatilize under nitrogen, but tend to leave behind a carbonaceous residue which subsequently is lost with the carbon black under oxygen. In order to accurately determine the liner composition, it is necessary to know the percent weight of carbonaceous residue found for each polymer in question. Table 65 summarizes data obtained from the literature and determined at Matrecon for some common membrane lining materials:

TABLE 65. CARBONACEOUS RESIDUE FOLLOWING PYROLYSIS UNDER N_2 AT 550°C

	Weight, %	
Polymer	Matrecon	Wake[a]
Neoprene	13.9	13.9
Polyvinyl chloride	4.9	5.9
Chlorosulfonated polyethylene	0.5	...
Butyl rubber	...	0.10

[a] Source: Wake, 1969, p 141.

Other corrections may be also applied for volatiles loss from fillers and/or carbon black and for CO_2 loss from a carbonate filler, e.g $CaCO_3$. These are relatively minor and are detailed in Chapter 8 of Wake (1969).

The experimental procedure used at Matrecon is outlined in Appendix N. Table 66 presents the membrane liner composition data obtained at Matrecon using the pyrolysis method. Ash results obtained in pyrolysis tests which use small specimens are compared with those obtained in the standard ASTM Test D297.

TABLE 66. ANALYSES OF POLYMERIC LINER MEMBRANES BY PYROLYSIS

Data are in Percent by Weight

Liner		Extractables		Carbon	Ash		
Polymer	Number[a]	+ polymer	Polymer	black	Pyrolysis	ASTM D297	Extractables[b]
Butyl rubber	22	57.9	...	21.5	20.6	...	...
	44	59.7	47.9	37.8	2.6	4.3	11.8
	57R	53.0	46.6	27.4	19.6	23.4	6.4
Chlorinated polyethylene	12	82.9	75.4	2.2	14.9	14.4	7.5
	77	81.6	72.5	2.8	15.6	12.6	9.1
	86	76.9	70.9	6.2	17.0	...	6.0
	99	59.2	...	37.5	3.2	...	...
Chlorosulfonated polyethylene	6R	47.3	43.6	45.9	6.9	3.3	3.8
	55	44.6	40.4	53.0	2.4	3.3	4.1
Ethylene propylene rubber	8	55.9	32.5	37.8	6.4	6.4	23.4
	16	64.7	...	31.1	4.3	...	...
	26	57.1	...	37.0	6.0	7.6	...
	36	94.6	...	5.4	0.0	0.9	...
	83R	53.5[c]	35.3[c]	47.1[c]	0.0[c]	0.3	18.2
	91	58.1	34.4	35.7	6.2	7.3	23.6
Neoprene	9	59.2	49.1	30.3	10.5	...	10.2
	43	59.2	45.5	29.6	11.1	12.2	13.7
	82	59.7	46.3	27.6	12.7	13.2	13.4
Polyester elastomer	75	95.6	92.9	6.4	-1.8	0.4	2.7
Polyvinyl chloride	11	86.2	52.3	6.3	7.5	6.2	33.9
	17	87.1	49.9	4.8	8.2	5.8	37.3
	59	93.4	57.5	2.2	9.3	6.9	35.9
	88	95.2	61.7	2.8	2.7	2.8	33.5
	93	90.1	57.8	6.4	3.6	...	32.3
	96	87.0	52.9	6.7	6.4	5.6	34.1
	97A	89.0	54.6	4.4	6.7	5.8	34.4

[a] Matrecon liner number; R = fabric-reinforced.
[b] Matrecon Test Method 2, Appendix M.
[c] Value based on one determination. All others based on duplicates.

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APPENDIX A
SUMMARY LIST OF THE EXPOSURES OF FLEXIBLE POLYMERIC MEMBRANE LINERS TO HAZARDOUS WASTES[a]

Polymer	Liner number	Type of compound	Nominal thickness, mils	Primary cells	Immersion	Weather		Pouches	Exposed to MSW leachate[b]
						Tub	Rack		
Butyl rubber	44	XL	62	...	2,4,5,7,9,10,11 14,15,18,19,20	...	x	...	x
	57R	XL	34	2,9,11,14	...	4	x	...	...
CPE	77	TP	30	2,5,9,11,14	2,4,5,7,9,10,11 14,15,18,19,20	2	x	19	x
	86	TP	22	...	...	...	...	2,5,7,9,10,19	x
	100	XL	36	...	2,4,5,7,10,11,14 15,16,18,19,20	...	...	...	...
CSPE	6R	TP	31	2,5,9,11,14	2,4,5,7,9,10,11 14,15,18,19,20	4,9	x	5,19	x
	55	TP	35	...	2,4,5,7,9,10,11 14,15,19,20	...	...	5,16,19	x
	85	TP	33	...	...	...	...	2,4,9	x
	125R	TP	36	17,18	...	...	...	...	...
ELPO	36	CX	20	2,4,5,9,10,11, 14,15,17,18,21	2,4,5,7,9,10,11 14,15,18,19,20	5,2	x	2,4,5,7,9,10, 11,14,15,16,19	...
EPDM	8	XL	62	...	...	2,9	x	...	x
	26	XL	26	2,9,11,14	...	...	x	...	x
	83R	TP	39	...	2,4,5,7,9,10,11 14,15,18,19,20	...	...	...	x
	91	XL	37	...	2,4,5,7,9,10,11 14,15,18,19,20	...	...	...	x
HDPE	105	CX	32	...	2,4,5,7,9,10,11, 14,15,16,18,19,20	...	...	...	...
LDPE	21	CX	10	...	...	...	...	5,7,10,16,19	x
	108	CX	31	...	2,4,5,7,9,10,11 14,15,16,18,19,20	...	...	...	...
Neoprene	43	XL	34	2,5,11,14,16, 17	...	...	x	...	x
	82	XL	60	...	...	5	x	...	x
	90	XL	37	...	2,4,5,7,9,10,11 14,15,18,19,20	...	...	...	x
Polybutylene	98	CX	8	...	...	...	...	2,4,5,7,10,11 14,15,16,19	x
Polyester	75	CX	7	2,5,9,11,14 15	2,4,5,7,9,10,11 14,15,18,19,20	5	x	...	x
Polypropylene	106	CX	33	...	2,4,5,7,9,10,11 14,15,16,18,19,20	...	...	...	...
PVC	11	TP	30	...	2,4,5,7,9,10,11 14,15,18,19,20	9	x	...	x
	17	TP	20	...	...	...	...	5,7,10,16,19	x
	19	TP	22	...	...	...	...	2,5,7,9,10,11 14,15,16,19	x
	59	TP	30	2,4,5,9,10, 11,14,15	2,4,5,7,9,10,11 14,15,18,19,20	5	x	19	x
	88	TP	20	...	2,4,5,7,9,10,11 14,15,18,19	...	...	2,7,9	x
	89	TP	11	...	20	...	...	...	x
	92	TP	20	18,21	18	...	...	...	x
	93	TP	11	...	...	...	...	7,5	x

[a] Code for wastes in exposure:
2 = spent caustic; 4 = slopwater; 5 = oil pond 104; 7 = weed oil; 9 = nitric acid waste; 10 = HF waste; 11 = pesticide; 14 = Pb waste; 15 = aromatic oil waste; 16 = Basin F; 17 = Well 118; 18 = deionized water; 19 = 5% NaCl solution; 20 = 0.1% tributyl phosphate; 21 = 50% Well 118/50% deionized water.

[b] Haxo et al, 1982, Table 42, page 125.

APPENDIX B

PROCEDURE
for
ANALYSIS AND CHARACTERIZATION OF WASTE LIQUIDS AND SLUDGES

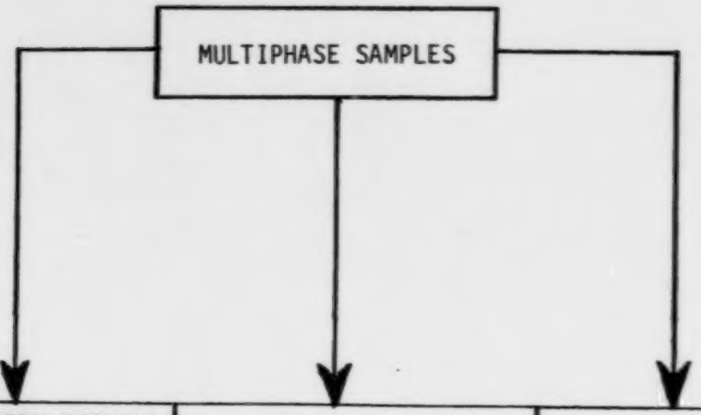
Used by

THE SANITARY ENGINEERING RESEARCH LABORATORY,
UNIVERSITY OF CALIFORNIA, BERKELEY, CA

in the
Analysis of Wastes

ANALYSIS AND CHARACTERIZATION OF WASTE LIQUIDS AND SLUDGES

Characterization Scheme



PHASE I. AQUEOUS INSOLUBLE ORGANIC LIQUID PHASE	PHASE II. AQUEOUS PHASE	PHASE III. SOLID PHASE
A. Flash point	A. Total solids and volatile solids	A. Flammability
B. Distillate at 95°C	B. pH, Total acidity and alkalinity	B. Percent inorganic and percent organic (ashing)
C. Viscosity	C. Water soluble volatile organics	C. Water extract
D. ASTM D2007-73 - Asphaltenes - Polar compounds - Saturated hydrocarbons - Aromatics	D. Oil and grease	
E. IR analysis (Chloroform extraction)	E. Lead analysis (AA)	
F. Water content	F. IR analysis of organics (Chloroform extraction)	

Note: 1. Separate a multiphase sample into Phases I, II, or III (as needed) using centrifugation or filtration. Report percent by weight of each phase present.

2. References to sources of test methods see Table 1.

PHASE I. AQUEOUS INSOLUBLE ORGANIC LIQUID PHASE

A. FLASH POINT

Determine flash point per ASTM D93-62 (closed cup) for fuel oils and viscous materials or ASTM D92 (open cup) for volatile flammable materials having flash point between 0-175°F.

B. DISTILLATE AT 95°C

If the organic phase is 10% by volume or higher, perform steam distillation up 95°C vapor temperature, noting volume of distillate. Multiply volume of distillate by 0.8 and report as percent by weight of organics boiling below 95°C. Record weight of organics boiling above 95°C as percent by weight. Obtain this value from organic phase remaining in distillation flask using volume times 0.8 to obtain weight percent.

C. VISCOSITY

Determine viscosity per ASTM D2983 at 10°C, 20°C, and 30°C.

D. ORGANIC GROUPS

Determine organic group content per ASTM D2007-69 chromatographic method. Report in percent by weight.

E. IR ANALYSIS

Chloroform extract.

F. WATER CONTENT OF PHASE I BY A TOLUENE AZEOTROPE METHOD (KOLTHOFF & SANDELL, 1943)

Weigh in a 250 mL stoppered round bottom distillation flask about 100 mL of PHASE I. Add glass beads and about 100 mL of toluene and swirl vigorously. Reflux the mixture for 30 minutes using a Henion design distillation head with finger type condenser. Collect the distillate at 85±2°C in a graduated separatory funnel. Shake the distillate and allow the phases to separate. Repeat until the aqueous layer is relatively clear. Read the volume of H_2O collected.

PHASE II. AQUEOUS PHASE

A. RESIDUE ON EVAPORATION, TOTAL SOLIDS, VOLATILE SOLIDS, AND SUSPENDED SOLIDS

In appropriate evaporating (nonmetallic) dish, evaporate 100 mL of solution on steam bath followed by drying, per current edition of "Standard Methods for the Examination of Water and Wastewater."

B. pH, TOTAL ACIDITY, AND ALKALINITY

Obtain pH of solution and measure acidity or alkalinity, per current edition of "Standard Methods for the Examination of Water and Wastewater."

C. WATER SOLUBLE VOLATILE ORGANICS

Distill a 200-mL volume of sample at 95°C vapor temperature. Record volume of organic liquid in distillate. Determine the density of the organic liquid. Record result as percent by weight water soluble volatile organics by multiplying the density by the volume.

D. GREASE

Place a sample of 500 mL - 1000 mL in a wide-mouth bottle. Extract the grease with hexane per Standard Methods (Soxhlet Extraction). Correct for residue left by hexane.

E. TOTAL LEAD IN AQUEOUS PHASE (API METHOD, pp. 747-763)

Pipette a 100 mL sample into a 400 mL beaker. Add 5 mL iodine solution and swirl to mix; add 20 mL of concentrated HNO_3, cover with a watchglass and heat to dryness. Dissolve the cooled residue in 20 mL of dilute HNO_3 and filter through a Whatman No. 42 filter paper, if necessary, into a 100 mL volumetric flask. Dilute to volume and aspirate into the atomic absorption instrument against aqueous lead standards. Carry a reagent blank with 100 mL of water.

PHASE III. SOLID PHASE

A. FLAMMABILITY

Place a small amount of dry solid phase on a spatula and place in upper part of burner flame (in hood); note if material burns, color and character of the flame, and if smoke or fumes are given off. Record observations.

B. PERCENT INORGANIC AND ORGANIC (ASHING)

Weigh 5.00 grams of dry solid phase in a porcelain crucible. Heat (in hood) to burn off majority of organics (if flammability test indicates a high organic content). Place crucible in electric furnace and heat at 550°C for one hour; cool and weigh. Report percent organic and inorganic content.

C. WATER EXTRACT

Weigh 20.00 grams of air-dry solid and add 100 mL of distilled water. Heat to boiling while stirring solution. Filter and allow filtrate to cool. On filtrate, determine pH and total residue on evaporation at 105°C. Report water soluble residue in mg/g of solids.

TABLE 1. METHODS FOR THE CHARACTERIZATION OF HAZARDOUS WASTES

	Phase[a]	
Liquid Organic	Aqueous	Solid
Flash point[b]	Total solids[c]	Flammability[d]
Distillate at 95°C[d]	Volatile solids[c]	Percent Organic[c]
Viscosity[b]	pH[d]	Percent Inorganic[c]
Organic groups[b]	Alkalinity or acidity[c]	Water Extract pH[c]
Water content[d]	Water soluble volatile organics[d]	Total Solids[c]
Others	Oil and grease[c]	Others
	Lead concentration[d]	
	Others	

[a] Separation of the three phases was made by centrifugation of the sample for about 40 minutes at 1,000 RPM.

[b] American Standards for the Testing and Materials.

[c] Standard Methods for the Examination of Water and Wastewater.

[d] Special Procedures of Sanitary Engineering Research Laboratories (SERL), University of California, Berkeley.

REFERENCES

American Petroleum Institute, Division of Refining. 1969. Manual of Disposal of Refinery Wastes, Volume 4. Washington, D.C.

American Public Health Association, American Water Works Association, and the Water Pollution Control Federation. 1975. Standard Methods for the Examination of Water and Wastewater, 14th edition.

American Society for Testing and Materials, Philadelphia, Pennsylvania.

Kolthoff, I. M. and E. B. Sandell. 1943. Textbook on Quantitative Inorganic Chemistry. MacMillan, New York.

Stephens, R. D. 1976. Hazardous Waste Sampling. In: Proceedings of Hazardous Waste Research Symposium. Residual Management by Land Disposal. EPA 600/9-76-015. U.S. Environmental Protection Agency. Cincinnati, Ohio. NTIS No. PB 256-595.

APPENDIX C

EAL CORPORATION REPORT DATED MARCH 13, 1981 ON THE CHEMICAL ANALYSIS OF FIVE WASTES

(GM/MS scans not included)

EAL CORPORATION
2030 Wright Avenue
Richmond, California 94804
(415) 235-2633
(TWX) 910-382-8132

March 13, 1981

Matrecon, Incorporated
Post Office Box 24075
Oakland, California 94623

Attention: Mr. Michael A. Fong

Reference: Purchase Order No. 1745
EAL Work Order No. 08300-000-0015

Dear Mr. Fong:

Enclosed please find our report for the five waste materials submitted under the referenced purchase order for chemical analysis and GC/MS.

The analyses were performed in accordance with standard waste analysis procedures as published by U.S. Environmental Protection Agency, and ASTM. The methods are referenced on each of the individual test reports. Our extraction procedure for the GC/MS study was modified somewhat from EPA's scheme, and is fully discussed in the report.

We applied our Quality Control Program to all phases of the analyses in order to assure the validity of the data. The program includes calibration curves, spikes, and blank determinations with each set of samples. A full description of the QC program is found in *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* U.S. EPA-600/4-79-019. QC data from this project, i.e., recovery, precision, and calibration information is available upon request.

Your billing for this report will be under separate cover. Your choice of EAL for this study is greatly appreciated. Please call me with any questions you may have concerning this report.

Sincerely,

B.W. Christensen

BEC/pv

Bent E. Christensen
Program Manager

Matrecon, Inc.
Oakland, CA

March 13, 1981

Work Order 08300-000-0015
Purchase Order No. 1745

CHARACTERIZATION ANALYSES FOR WASTE MATERIALS-GENERAL PHYSICAL TESTS

		Lead Waste 1708-1-3	Spent Caustic 1701-1-5	Pesticide Waste 1708-1-2
1. Description				
Form		Dark brown liquid with suspended and floating solids.	Cloudy, yellow liquid with white crystalline solids at bottom	Cloudy, white liquid; milky appearance
Phases		Separates by centrifugation into light brown aqueous phase with dark brown oil on top and dark brown lumps floating on oil.*	Aqueous phase with solids.	Aqueous.
Odor		Strong gasoline odor.	Odorless	Weak phenol and chloro-naphtalene odor.
2. Specific Gravity	Aqueous Phase:	0.97 g/mL	1.19 g/mL	1.00 g/mL
(includes suspended solids)	Organic phase:	0.79 g/mL	---	---
3. Organic/Inorganic/Water Ratio (by weight)		13.6/0.19/86.2	3.4/23.5/73.1	0.24/0.41/99.35
Supporting Data				
a) Total solids in aquous phase,% w/w		0.35	26.9	0.65
b) Non-volatile solids at 550°C, in aquous phase,% w/w		0.19	23.5	0.41
c) Water in organic phase (Karl Fisher),%, w/w				
d) Total Organic Carbon (TOC) %, w/w			1.07	0.071
4. pH		7.6	7.7	2.6
5. Flash Point, °F		<32	Boils without flashing	Boils without flashing

* Phase ratio (aquous/oil): By Volume: 84.2/5.8; By Weight: 86.4/13.6

continued

Matrecon, Inc.
Oakland, CA

March 13, 1981

Work Order 08300-000-0015
Purchase Order No. 1745

Page 2 - Characterization Analyses for Waste Materials-General Physical Tests

	Oil Pond 104 Waste 1701-1-4	Strong Acid Waste 1705-1-1
1. Description		
Form	Dark brown-black oil of tar-like consistency.	Clear, yellow liquid.
Phases	Single organic phase.	Aquous
Odor	Odor as used automobile oil.	Weak with acid odor.
2. Specific Gravity:	Aquous phase:	1.07 g/mL
(includes suspended solids)	Organic phase: 0.92 g/mL	---
3. Organic/Inorganic/Water Ratio (by weight)	99.0/0.39/0.61	2.62/1.12/96.26
Supporting data		
a) Total solids in aquous phase %, w/w	---	3.74
b) Non-volatile solids at 550°C in aquous phase, % w/w	---	1.12
c) Water in organic phase (Karl Fisher), % w/w	0.61	---
d) Total Organic Carbon (TOC) %, w/w		0.78
4. pH	---	0.5
5. Flash point, °F	Boils without flashing	Boils without flashing.

Matrecon, Inc,
Oakland, CA

March 11, 1981
W.O. 08300-000-0015
P.O. No. 1745

HEAVY METALS BY ATOMIC ABSORPTION

ANALYSIS RESULTS

mg/liter

	Lead Waste						Limits of Detection	
	Aquous Phase 1708-1-3	Organic Phase	Spent Caustic 1708-1-5	Pesticide Waste 1708-1-2	Oil Pond 104 Waste 1708-1-4	Strong Acid Waste 1708-1-1	Aquous	Organic
Antimony	9.0	500	50	9.0	500	9.6		
Arsenic	0.02	0.02	N.D.	N.D.	N.D.	0.19	0.02	0.02
Beryllium	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	0.2	0.4
Cadmium	N.D.	4.7	0.6	N.D.	8.0	0.22	0.15	
Chromium	1.2	5.7	N.D.	5.4	286	22	0.8	
Copper	0.46	4.8	2.3	9.2	33	5.6		
Lead	13	530	5.0	1.4	170	28		
Mercury	0.02	0.26	N.D.	N.D.		N.D.	0.003	
Nickel	0.9	0.9	1.8	24.5	4.0	9.0		
Selenium	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1.0	1.0
Silver	N.D.	0.2	0.33	N.D.	11.5	N.D.	0.05	
Thallium		N.D.	0.03	N.D.	N.D.	0.21	0.02	0.02
Zinc	74	11	8.2	150	450	42		
Barium	4.8	N.D.	19	4.8	N.D.	5.0		5
Cobalt	1.2	43	24	1.2	40	2.9		
Molybdenum	0.5	0.1	2.0	0.5	0.1	0.8		
Vanadium	N.D.	3.5	5.1	N.D.	10.1	N.D.	2.5	
Titanium	3.0	38	12	3.0	230	19		
Organolead	200		--	--	--	--		
Boron	9.6		--	26	--	--		

Matrecon, Inc.
Oakland, California

March 13, 1981
P.O. 1745
W.O. 08300-0015

CLASSIFICATION OF ORGANIC CONSTITUENTS

I. Functional Group Breakdown (ppm w/v):

	Lead Waste 1708-1-3	Oil Pond 104 Waste 1708-1-4	Strong Acid Waste 1708-1-1
Alkanes	5000	11700	< 4
Oxygenates	100	100	< 4
Olefins and Aromatic Hydrocarbons	25200	1500	60

Method: ASTM D 1019-58T; GC-FID & Electrol.Cond. - OV101 & FFAP.

II. Polarity Ranges (log p*), ppm w/v:

Lead Waste 1708-1-3		Pesticide Waste 1708-1-2		Oil Pond 104 Waste 1708-1-4		Reference Table	
Fraction ppm	log p	Fraction ppm	Log p	Fraction ppm	Log p	Compound	Log p
2.6	3.93	1.3	3.93	6900	3.53	Acetone	0.55
11.8	4.62	0.66	4.46	16000	3.93	Benzene	2.13
15.6	5.43	1.2	5.07	41000	4.68	Naphthalene	3.37
9.8	6.27	0.88	5.87	31000	5.43	Diphenyl Ether	4.20
2.6	7.22	0.38	6.80	15000	6.27	4,4' - PCB	5.58
0.26	7.94	0.29	7.81	4400	7.20	p,p' - DDT	6.19
0.52	8.69	0.28	8.87	980	7.94	Arochlor 1254	6.72
				93	8.32		

* log p = log 9n-octanol/water partition coefficient)

Method: Fed Reg. 43, No. 243, p. 58966, App. XI; HPLC - Micro Bondapak C_{18}.

Matrecon, Inc
Oakland, California

March 13, 1981
P.O. 1745
W.O. 08300-0015

HAZARDOUSNESS CHARACTERISTICS
REACTIVITY

	Lead Waste 1708-1-3	Spent Caustic 1708-1-5	Pesticide Waste 1708-1-2	Oil Pond Waste 1708-1-4	Strong Acid Waste 1708-1-1
I. Reaction With:					
Water -	No reaction (miscible)	No reaction (miscible)	No reaction (miscible)	No reaction (immiscible)	No reaction (miscible)
Acid -	Forms dark brown precipitate	Turns clear and yellow	Forms white precipitate	No reaction (immiscible)	No reaction (miscible)
Alkali -	Forms dark brown precipitate	Forms white precipitate	Forms yellow to brown precipitate	No reaction (immiscibel)	Forms yellow precipitate with generation of heat

Method: Fed. Reg. 45, No. 98, p. 33122, § 261.23

	Lead Waste 1708-1-3	Spent Caustic 1708-1-5	Pesticide Waste 1708-1-2	Oil Pond Waste 1708-1-4	Strong Acid Waste 1708-1-1
II. Toxic Gas Evolution:					
H_2S -	N.D.	N.D.	N.D.	N.D.	N.D.
HCN -	0.2 ppm	0.8 ppm	N.D.	0.1 ppm	1.8 ppm

Method: Ibid and unpublished communication. EPA Washington, 1980.

N.D. = None Detected

EAL CORPORATION
2030 Wright Avenue
Richmond, California 94804
(415) 235-2633
(TWX) 910-382-8132

ANALYSIS REPORT

Customer: Matrecon, Incorporated
Mr. Mike Fong
Post Office Box 24075
Oakland, California 94623

Date: March 11, 1981
Samples Received: January 14, 1981
EAL W. O. No. 08300-000-0015
Purchase Order No.: 1745

GC/MS Analysis of Hazardous Waste Samples

Sample Preparation

Three samples were submitted for GC/MS analyses:

Dismantling Pest. Waste	EAL No. 1708-1-2
Lead Waste	1708-1-3
Oil Pond 104	1708-1-4

All three samples were adjusted to pH 11 with sodium hydroxide (NaOH) and extracted with methylene chloride (CH_2Cl_2). The remaining solution was then acidified to pH 2 with sulfuric acid (H_2SO_4) and extracted with CH_2Cl_2. This is a standard procedure for the separation of samples into base/neutral and acidic extractable fractions prior to analysis for the organic priority pollutants. (EPA Method 625). After extraction, the solvent was evaporated and the sample rediluted with one mL of CH_2Cl_2.

The acid extractable fractions from all three samples and the base/neutral fraction of the dismantling pest. waste (1708-1-2) were relatively clean and free of oils and other materials that would prevent their analysis. Internal standard was added to these fractions and they were analyzed without any further cleanup.

The base/neutral fractions of the lead waste (1708-1-3) and oil pond 104(1708-1-4) contained large amounts of oil and other contaminants which EAL knows from experience would make analysis impossible. These samples had to be processed further before any analysis could be made. The oil and other material were removed from these fractions by passing them through an alumina column and eluting the column with four 50 mL volumes of hexane, benzene, and chloroform.

B.E. Christensen
Bent E. Christensen
Program Manager

EAL is an approved State of California chemical, radiological, bacteriological and fish toxicity bioassay laboratory; an accredited American Industrial Hygiene Association laboratory; and a licensed clinical laboratory.

The hexane fraction contained oil and was not analyzed. The benzene and first chloroform fractions were free from oil and were evaporated and rediluted to 1 mL with CH_2Cl_2 and then analyzed after addition of internal standard. The fourth fraction was not analyzed.

Analytical Methods:

The analyses were performed using a 30 meter SE-30 fused silica capillary column (0.021 mm I.D.) connected directly to the source of the mass spectrometer. The column was temperature programmed from 50°C to 300°C @ 8°C/min. 1.0 microliters of each sample were injected onto the column using splitless injection. The mass spectrometer was scanned from m/z 25 to 450 in one second intervals. All the data was stored on fixed discs. The amounts of the priority pollutants were calculated from comparison to the internal standard using response factors created from standard runs. The amounts of compounds similar in chemical structure to the priority pollutants were calculated manually by using a response factor from the similar priority pollutant and the area of the internal standard. All compounds were identified using a computer library search.

Results and Discussion:

Because of the extremely complex nature of the samples, not all individual components could be quantitated. Those that were are subject to error because the solvent extraction and alumina column cleanup recoveries are uncertain and the response factors used for compounds other than priority pollutants are approximate. In general, many of the compounds in the samples are chemically related to the priority pollutants and in some cases, make up the bulk of the sample. Significant differences in the chemical nature of the samples can be seen when the percentages of the different classes of compounds in each sample are compared.

Dismantling Pest. Waste (1708-1-2):

143 compounds were detected in this sample. Phenols accounted for 21% of the acid fraction and their distribution is given in Table I. The other major components of the sample were alcohols, ketones and some aldehydes.

5-ethyl-2-methylheptane accounted for 71% of the base neutral fraction. The amounts of the base/neutral priority pollutants found are given in Table I.

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Lead Waste (1708-1-3):

313 compounds were detected in this sample. The acid fraction contained 58% oxepane (a seven membered ring containing oxygen), 19% diisooctylphthalate and 7% phenols. The distribution of the phenols is given in Table II. The remainder of the sample contained small percentages of aromatic and aliphatic acids.

The base neutral fraction contained 12% substituted benzene; 3% naphthalenes; 10% phenanthrenes; 3% Fluorenes; 9% 1,3-oxathiolane and 27% phthalates. The priority pollutants found are listed in Table II.

Oil Pond (1708-1-4):

336 compounds were detected in this sample. The acid fraction contained 50% phenols, 14% benzoic acids and 13% aliphatic acids. The distribution of the phenols is given in Table III.

The base/neutral fraction contained 34% phenanthrenes.. Phenanthrene itself accounted for approximately 3% of the sample. The priority pollutants found in this sample are given in Table IV.

Summary:

Although the qualitative identification and quantitative estimates of certain individual compounds may be subject to error, the data clearly indicates that the samples contain significant amounts of organic compounds which are chemically similar to the 129 priority pollutants listed in the Federal Register. These include phenols, phthalates, naphthalenes, and phenanthrenes.

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TABLE I

DISMANTLING PEST. WASTE
(1708-1-2)

Acid Fraction

Compound	Concentration (ppm)
2-chlorophenol	3
2,6-dichlorophenol	4
2,3-dichlorophenol	2
2-chloro-6-methylphenol	1
4-(1,1-dimethylethyl) phenol	2
trichlorophenol	15
trichlorophenol	20
trichlorophenol	78
2,5-dichlorophenol	78
2,3-dichlorophenol	57

Base Neutral Fraction

Compound	Concentration (ppm)
n-nitrosodiphenylamine	3
di-n-butylphthalate	8
butyl-benzylphthalate	3

TABLE II

LEAD WASTE
(1708-1-3)

Acid Fraction

Compound	% of Total Sample	(ppm) Approximate Concentration
2-methylphenol	0.72	33
2,6-dimethylphenol	0.5	23
2-ethylphenol	0.26	12
3,4-dimethylphenol	0.68	32
2,3-dimethylphenol	0.94	44
2,3,5-trimethylphenol	0.18	8
2-ethyl-5-methylphenol	0.21	10
4-ethyl-2-methylphenol	0.25	11
2-ethyl-4-methylphenol	0.70	32
2,3,5-trimethylphenol	0.95	45
2,4,5-trimethylphenol	1.17	55
2,4-dimethylphenol	1.5	74

Base/Neutral Fraction

Compound	(ppm) Approximate Concentration*
Anthracene	14
di-m-butylphthalate	1
fluoranthene	9
butylbenzylphthalate	1.5
chrysene	4
benzo(a)pyrene	3.4

* weak spectra obtained - tentative identification and quantitation.

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Matrecon, Inc.
Oakland, CA

March 11, 1981

W.O. 08300-000-0015
P.O. No. 1745

TABLE III

OIL POND 104
(1708-1-4)

Acid Fraction

Compound	Approximate Concentration (ppm)
phenol	85
2-methylphenol	67
4-methylphenol	328
2,6-dimethylphenol	7
2-ethylphenol	35
2,3-dimethylphenol	248
2,4-dimethylphenol	364
2,5-dimethylphenol	90
3,4-dimethylphenol	127
2-(1-methylethyl)phenol	16
4-ethyl-2-methylphenol	41
4-ethyl-3-methylphenol	42
2-ethyl-4-methylphenol	142
2,3,5-trimethylphenol	46
2,4,5-trimethylphenol	64
4-ethyl-5-methylphenol	29
3-(1,1-dimethylethyl)-phenol	63
5-methyl-2-(1-methylethyl)phenol	17

Matrecon, Inc.
Oakland, CA

March 11, 1981

W.O. 08300-000-0015
P.O. No. 1745

TABLE IV

OIL POND 104
(1708-1-4)

Base/Neutral Fraction

Compound	Approximate Concentration (ppm)
naphthalene	4
fluorene	4
phenanthrene	185
anthracene	19
di-n-butylphthalate	13
pyrene	6
butylbenzylphthalate	13
chrysene	117
benzo(a)anthracene	106
n-nitrosodiphenylamine	17

APPENDIX D
MAJOR EXTRACTABLE ORGANICS RECOVERED FROM WASTES DETERMINED BY GAS CHROMATOGRAPHY AND MASS SPECTROSCOPY (GC/MS)

Lead waste		Pesticide waste		Oil Pond 104	
Compound	ppm	Compound	ppm	Compound	ppm
2,2,4-trimethyl oxepane	2500	5-ethyl-2-methyl heptane	1700	Dichloromethane	450
Diisooctylphthalate	800	Trichlorophenol(s)	113	2,4-dimethylphenol	360
2,4-dimethylphenol	74	1-(2-chlorophenyl) ethanol	87	4-(1,1-dimethylethyl) benzoic acid	340
1,3-oxathiolane	62	2,5-dichlorophenol	78	4-methylphenol	330
Benzeneacetic acid	59	2,7-dimethyl naphthalene	76	2,3-dimethylphenol	250
Phenanthrene caboxylic acid	55	Pentafluorophenol	74	Diisooctylphthalate	190
2,4,5-trimethyl phenol	55	1,3-oxathiolane	66	Phenanthrene(s)	190
2,3,5-trimethyl phenol	45	2,3-dichlorophenol	59	2-ethyl-4-methyl phenol	140
2,3-dimethylphenol	44	2-butoxyethanol	51	3-methylcinnoline	140
2-methylphenol	33	1-(2-butoxyethoxy) ethanol	50	3,4-dimethylphenol	127
3,4-dimethylphenol	32	Dibutyphthalate	8	Chrysene	117
2-ethyl-4-methyl phenol	32	2,6-dichlorophenol	4	Benzo(a)anthracene	106
2,6-dimethylphenol	23	2-chlorophenol	3	2,5-dimethylphenol	90
3-methylphenanthrene	21	N-nitroso diphenylamine	3	Phenol	85
Anthracene	14	Butylbenzylphthalate	3	2-methylphenol	67
2-ethylphenol	12	4-(1,1-dimethylethyl) phenol	2	2,4,5-trimethyl phenol	64
4-ethyl-2-methyl phenol	11	2-chloro-6-methyl phenol	1	3-(1,1-dimethylethyl) phenol	63
2-ethyl-5-methyl phenol	10			2,3,5-trimethyl phenol	46
Fluoranthene	9			4-ethyl-3-methyl phenol	42
Chrysene	4			2-ethyphenol	35
Benzo (a) pyrene	3.4			4-ethyl-5-methyl phenol	29
Butylbenzylphthalate	1.5			Anthracene	19
Dibutylphthalate	1			N-nitroso diphenylamine	17
				2-methyl-2-(1-methylethyl) phenol	7
				Dibutylphthalate	13
				Butylbenzylphthalate	13
				2,6-dimethylphenol	7
				Pyrene	6
				Naphthalene	4
				Fluorene	4

APPENDIX E

MATERIALS FOR SOIL AND ADMIX LINERS

Material		Supplier
Soil		
Fine-grain, high silica content		Mare Island Naval Shipyard, Vallejo, California
Soil cement		
Soil:	"Waste fines" from rock crushing	Quarry Products Company, Richmond, California
Cement:	Type 1-2-5 Portland cement	Kaiser Permanente
	Rice Hull ash (RHA) cement	Industrial Materials Co., Los Angeles, California
Coating C:	Coal-tar epoxy, Ceilcote Flake Prime and Flake Tar	Ceilcote
Coating K:	Coal-tar epoxy, Bitumastic 300M	Koppers
Hydraulic asphalt concrete (HAC)		
Hot mix		Ransome Co., Emeryville, California
Aggregate, dense-graded 0.25-in. max.		Lone Star Quarry, Livermore, California
Asphalt, AR-4000		Douglas Oil Company
Modified bentonite		
Sand, dense-graded No. 2 (contained 1% water)		Topsoil King, Richmond, California

APPENDIX F

PROPERTIES OF UNEXPOSED POLYMERIC MEMBRANES[a]

Polymer[b]		Butyl	Butyl	CPE	CPE	CPE	CSPE
Compound type[d]		XL	XL	TP	TP	XL	TP
Fabric, type		...	Nylon	...	...	...	Nylon
Thread count, epi		...	22x11	...	...	...	8x8
Nominal thickness, mil		62.5	34	30	22	36	31
Matrecon liner serial number[e]		44	57R	77	86	100	6R
	Direction of test						
Analytical properties:							
Specific gravity		1.176	1.286	1.362	1.377	1.390	1.343
Ash (db)[f], %		4.28	23.46	12.56	17.37	6.02	3.28
Volatiles, %		0.46	0.29	0.14	0.05	0.66	0.51
Extractables (db)[f], %		11.79	6.36	9.13	6.02	17.42	3.77
Solvent[g]		MEK	MEK	n-heptane	n-heptane	n-heptane	DMK
Physical properties:							
Average thickness, mil		62	34	29	22	36	31
Tensile at fabric break, ppi	Machine	...	73.1	...	...	...	37.7
	Transverse	...	72.3	...	...	...	34.0
Elongation at fabric break, %	Machine	...	25	...	...	...	30
	Transverse	...	25	...	...	...	15
Tensile at ultimate break, psi	Machine	1625	h	2055	1845	1880	1845
	Transverse	1570	h	2340	1510	1935	1610
Tensile at ultimate break, ppi	Machine	104.1	h	59.6	40.6	67.6	59.7
	Transverse	100.2	h	66.7	34.1	69.6	52.5
Elongation at break, %	Machine	415	60	325	355	460	245
	Transverse	470	25	480	595	400	240
Tensile set, %	Machine	18	4	140	208	43	97
	Transverse	18	2	160	235	33	93
S-100, psi	Machine	335	...	1240	870	555	995
	Transverse	280	...	560	275	680	880
S-100, ppi	Machine	21.4	...	36.0	19.1	20.0	32.2
	Transverse	17.9	...	16.0	6.2	24.4	28.7
S-200, psi	Machine	750	...	1540	1210	1295	1710
	Transverse	615	...	820	405	1455	1390
S-200, ppi	Machine	48.0	...	44.7	26.6	46.5	55.4
	Transverse	39.2	...	23.4	9.2	52.3	45.3
Tear strength (Die C), lb	Machine	12.88	...	7.83	4.05	10.58	...
	Transverse	14.05	...	6.93	3.91	10.68	...
Tear strength (Die C), ppi	Machine	201	...	273	187	297	...
	Transverse	221	...	239	178	304	...
Puncture resistance:							
Thickness, mil		62	34	29	22	35	34
Stress, lb		39.5	26.6	43.9	20.9	40.0	33.7
Elongation, in.		1.17	0.26	0.94	0.91	0.95	0.59
Hardness, Durometer points		54A	71A	80A	67A	63A	77A

Continued

APPENDIX F (Continued)

Polymer[b]		CSPE	CSPE	CSPE	ELPO	EPDM	EPDM	EPDM
Compound type[d]		TP	TP	TP	CX	XL	XL	TP
Fabric, type		...	...	Polyester	...	...	...	Polyester
Thread count, epi		...	...	8x8	...	...	...	8x8
Nominal thickness, mil		35	33	30	22	62.5	30	40
Matrecon liner serial number[e]		55	85	125R	36	8	26	83R
	Direction of test							
Analytical properties:								
Specific gravity		1.371	1.311	1.296	0.938	1.173	1.169	1.199
Ash (db)[f], %		3.32	4.02	3.99	0.90	6.78	7.67	0.32
Volatiles, %		0.42	0.92	0.12	0.15	0.38	0.50	0.31
Extractables (db)[f], %		4.08	8.22	8.97	5.50	23.41	22.96	18.16
Solvent[g]		DMK	DMK	DMK	MEK	MEK	MEK	MEK
Physical properties:								
Average thickness, mil		35	33	29	23	62	36	39
Tensile at fabric break, ppi	Machine	...	...	53.4	...	...	...	43.2
	Transverse	...	...	41.4	...	...	...	29.0
Elongation at fabric break, %	Machine	...	...	19	...	...	...	20
	Transverse	...	...	33	...	...	...	...
Tensile at ultimate break, psi	Machine	1860	2345	...	2715	1635	1935	1010
	Transverse	1565	2055	...	2525	1550	1865	870
Tensile at ultimate break, ppi	Machine	65.0	75.0	53.0	61.0	98.9	74.5	39.7
	Transverse	55.1	66.2	46.6	55.6	94.9	70.9	34.8
Elongation at break, %	Machine	260	260	220	675	520	440	265
	Transverse	300	325	245	655	500	460	240
Tensile set, %	Machine	75	167	73	465	14	9	59
	Transverse	97	192	83	445	11	9	51
S-100, psi	Machine	1110	1150	...	940	350	385	890
	Transverse	650	750	...	905	320	330	730
S-100, ppi	Machine	38.9	36.8	41.8	21.1	21.2	14.8	35.0
	Transverse	23.1	24.2	29.0	19.9	19.6	12.5	29.2
S-200, psi	Machine	1810	2130	...	1035	800	925	990
	Transverse	1205	1410	...	1000	740	830	845
S-200, ppi	Machine	63.3	68.2	51.8	23.2	48.4	35.6	38.9
	Transverse	42.5	66.2	42.5	22.0	45.3	31.5	33.8
Tear strength (Die C), lb	Machine	10.31	9.77	...	8.90	12.7	7.33	...
	Transverse	9.57	8.78	...	8.23	12.8	7.47	...
Tear strength (Die C), ppi	Machine	294	308	...	388	206	193	...
	Transverse	271	277	...	369	211	197	...
Puncture resistance:								
Thickness, mil		35	33	28	22.5	60	37	39
Stress, lb		45.0	47.8	30.6	26.3	56.9	31.3	33.6
Elongation, in.		0.83	0.86	0.61	0.97	1.46	1.24	0.61
Hardness, Durometer points		78A	79A	75A	89A	57A	58A	70A
Hardness, Durometer points		32D	...	28D	32D	...	...	...

Continued

APPENDIX F (Continued)

Polymer[b]		EPDM	HDPE	HDPE[c]	LDPE[c]	LDPE[c]	Neoprene	Neoprene
Compound type[d]		XL	CX	CX	CX	CX	XL	XL
Fabric, type		...	...	...	...	...	...	...
Thread count, epi		...	...	...	...	...	...	...
Nominal thickness, mil		31	100	31	10	31	31	62.5
Matrecon liner serial number[e]		91	99	105	21	108	43	82
	Direction of test							
Analytical properties:								
Specific gravity		1.160	0.943	0.948	0.931	0.921	1.477	1.480
Ash (db)[f], %		7.33	0.10	0.03	0.00	0.04	12.30	13.21
Volatiles, %		0.34	0.06	0.14	0.09	0.18	0.45	0.19
Extractables (db)[f], %		23.64	...	0.00	3.60	2.07	13.69	13.43
Solvent[g]		MEK	...	MEK	MEK	MEK	DMK	DMK
Physical properties:								
Average thickness, mil		37	103	32	9	31	33	61
Tensile at yield, psi	Machine	...	2715	3745	1490	1455	...	...
	Transverse	...	2640	3815	1175	1455	...	...
Tensile at yield, ppi	Machine	...	306.5	118.4	14.2	41.6	...	...
	Transverse	...	291.9	122.9	10.7	41.8	...	...
Tensile at break, psi	Machine	1865	2185	2610	2990	2085	1910	1835
	Transverse	1790	2195	2355	2940	1975	1660	1675
Tensile at break, ppi	Machine	67.2	246.5	81.3	28.4	59.7	65.9	113.8
	Transverse	66.3	231.4	75.8	26.8	56.6	56.0	100.2
Elongation at break, %	Machine	475	750	100	510	535	330	390
	Transverse	500	675	125	675	575	310	410
Tensile set, %	Machine	11	640	85	395	435	8	10
	Transverse	10	585	107	535	470	6	9
S-100, psi	Machine	375	1965	2635	1490	1375	490	405
	Transverse	300	1920	2385	1175	1265	430	360
S-100, ppi	Machine	13.5	221.7	82.2	14.2	39.4	16.9	25.1
	Transverse	11.2	212.3	74.7	10.7	36.2	14.5	21.5
S-200, psi	Machine	915	1980	...	1610	1385	1105	875
	Transverse	795	1945	...	1165	1300	970	705
S-200, ppi	Machine	32.9	223.5	...	15.3	39.5	38.1	54.3
	Transverse	29.5	215.2	...	10.6	37.2	32.7	42.2
Modulus of elasticity, psi	Machine	...	78,600	150,150	19,400	21,960	...	...
	Transverse	...	78,700	158,750	24,400	24,870	...	...
Tear strength (Die C), lb	Machine	7.27	...	40.17	4.07	14.96	5.40	11.57
	Transverse	7.16	...	36.00	3.54	13.91	5.43	10.70
Tear strength (Die C), ppi	Machine	196	...	1215	420	516	171	183
	Transverse	195	...	1110	365	479	170	178
Puncture resistance:								
Thickness, mil		37	99	32	9.6	31	33	60
Stress, lb		29.2	131.0	51.2	13.7	33.5	30.6	53.9
Elongation, in.		1.17	0.33	0.25	0.79	0.51	1.14	1.29
Hardness, Durometer points		52A	95A	90A	86A	93A	57A	57A
Hardness, Durometer points		...	59D	60D	41D	38D	...	...

Continued

APPENDIX F (Continued)

Polymer[b]	Direction of test	Neoprene	Polybutylene[c]	Polyester	Polypropylene[c]	PVC	PVC
Compound type[d]		XL	CX	CX	CX	TP	TP
Fabric, type		...	...	...	...	...	...
Thread count, epi		...	...	...	...	...	...
Nominal thickness, mils		37	8	7	33	30	20
Matrecon liner serial number[e]		90	98	75	106	11	17
Analytical properties:							
Specific gravity		1.390	0.915	1.236	...	1.276	1.254
Ash (db)[f], %		4.67	0.08	0.38	...	6.14	5.81
Volatiles, %		0.37	0.12	0.26	0.01	0.15	0.44
Extractables (db)[f], %		21.46	...	2.74	...	33.90	34.11
Solvent[g]		DMK	...	MEK	...	$CCl_4 + CH_3OH$	
Physical properties:							
Average thickness, mil		37	8	7	33	30	20
Tensile at yield, psi	Machine	...	...	...	5015	...	...
	Transverse	...	...	...	5020	...	...
Tensile at yield, ppi	Machine	...	...	...	162.5	...	...
	Transverse	...	...	...	160.9	...	...
Tensile at break, psi	Machine	2185	5625	6770	[i]	3005	2910
	Transverse	2010	5580	6765	3035	2750	2675
Tensile at break, ppi	Machine	80.9	42.8	47.4	[i]	90.2	56.7
	Transverse	74.4	44.6	47.4	99.5	82.5	52.2
Elongation at break, %	Machine	415	390	560	40	350	350
	Transverse	415	375	590	75	365	365
Tensile set, %	Machine	26	346	340	16	91	70
	Transverse	25	331	370	50	106	83
S-100, psi	Machine	565	2330	2715	...	1495	1360
	Transverse	550	2360	2455	3055	1345	1180
S-100, ppi	Machine	21.0	17.7	19.0	...	44.9	26.5
	Transverse	20.4	18.9	17.2	100	40.4	23.0
S-200, psi	Machine	1450	3035	2880	...	2140	1915
	Transverse	1225	3200	2585	...	1885	1690
S-200, ppi	Machine	53.7	23.1	20.2	...	64.2	37.3
	Transverse	45.3	25.6	18.1	...	56.6	33.0
Modulus of elasticity, psi	Machine	...	...	...	190,900	...	...
	Transverse	...	...	...	184,300	...	...
Tear strength (Die C), lb	Machine	7.74	2.61	6.38	12.25	11.37	6.56
	Transverse	7.29	2.85	5.47	9.37	11.04	5.94
Tear stength (Die C), ppi	Machine	207	355	911	393	379	332
	Transverse	196	380	782	302	368	301
Puncture resistance:							
Thickness, mil		37	7.5	7.8	33	31	20
Stress, lb		44.9	13.9	29.9	60.3	38.6	25.30
Elongation, in.		1.01	0.66	1.30	0.65	0.64	0.70
Hardness, Durometer points		61A	94A	93A	...	80A	76A
Hardness, Durometer points		...	...	49D	68D	...	29D

Continued

APPENDIX F (Continued)

Polymer[b]		PVC	PVC	PVC	PVC	PVC	PVC
Compound type[d]		TP	TP	TP	TP	TP	TP
Fabric, type		-	-	-	-	-	-
Thread count, epi		-	-	-	-	-	-
Nominal thickness, mil		20	30	20	10	20	10
Matrecon liner serial number[e]		19	59	88	89	92	93
	Direction of test						
Analytical properties:							
Specific gravity		1.231	1.280	1.255	1.308	...	1.283
Ash (db)[f], %		3.65	6.97	2.80	5.67	5.84	4.94
Volatiles, %		0.05	0.31	0.17	0.03	0.06	0.12
Extractables (db)[f], %		38.91	35.86	33.46	25.17	32.75	32.26
Solvent[g]		$CCl_4 + CH_3OH$					
Physical properties:							
Average thickness, mil		22	33	20	11	20	11
Tensile at break, psi	Machine	2495	2685	3395	3715	2435	3575
	Transverse	2335	2430	2910	3085	2145	3035
Tensile at break, ppi	Machine	52.2	87.5	67.9	40.9	48.7	38.1
	Transverse	49.0	79.2	58.2	33.9	42.9	33.4
Elongation at break, %	Machine	310	355	325	315	245	325
	Transverse	340	395	335	325	255	350
Tensile set, %	Machine	55	45	102	195	43	98
	Transverse	71	56	101	205	48	117
S-100, psi	Machine	1410	1020	1870	1845	1515	1750
	Transverse	1250	970	1600	1530	1365	1420
S-100, ppi	Machine	29.5	33.3	37.4	20.3	30.3	18.7
	Transverse	26.3	31.6	36.0	16.8	27.3	15.6
S-200, psi	Machine	1935	1715	2610	2715	2170	2580
	Transverse	1675	1445	2190	2195	1885	2055
S-200, ppi	Machine	40.5	55.9	52.2	29.9	43.4	27.5
	Transverse	35.1	47.1	43.8	24.1	37.7	22.6
Tear strength (Die C), lb	Machine	6.49	10.25	9.26	4.49	8.70	4.26
	Transverse	6.05	9.54	9.17	4.30	7.46	3.99
Tear strength (Die C), ppi	Machine	295	313	463	408	435	400
	Transverse	275	290	470	391	373	362
Puncture resistance:							
Thickness, mil		22	32	20	11	20	11
Stress, lb		24.0	40.0	28.6	17.8	27.4	15.9
Elongation, in.		0.71	0.75	0.56	0.48	0.62	0.55
Hardness, Durometer points		72A	73A	80A	82A	82A	78A
		...	26D	...	...	30D	...

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[a] Methods used for determining properties of unexposed polymeric membranes; ASTM D297, Method A for specific gravity; ASTM D297 for ash; volatiles was defined as percent weight loss after 2 hours at 105°C; Matrecon Test Method No. 2 for extractables (Matrecon, 1983, pp 340-43); ASTM D412 for tensile properties using the Type IV test specimen; ASTM D638 for tear strength; ASTM D882 modified, for modulus of elasticity; FTMS 101B, Method 2065 for puncture resistance; and ASTM D2240 for hardness. Note that all tensile and tear testing reported in this appendix was done at 20 ipm.

[b] CPE = Chlorinated polyethylene; CSPE = chlorosulfonated polyethylene; ELPO = elasticized polyolefin; EPDM = ethylene propylene rubber; PVC = polyvinyl chloride.

[c] Unpigmented, i.e. compounded without a filler.

[d] XL = Crosslinked; TP = Thermoplastic; CX = Crystalline.

[e] Contractors' identification number. R = Fabric-reinforced.

[f] db = Dried basis.

[g] MEK = Methyl ethyl ketone; DMK = dimethyl ketone = acetone; $CCl_4 + CH_3OH$ = 2:1 blend of carbon tetrachloride and methyl alcohol.

[h] Bulk of lining materials' strength is in the nylon fabric. The butyl coating over the fabric tended not to fail catastrophically, and no useful value could be obtained for tensile strength at ultimate break.

[i] Sheeting tended to fail after yielding and no value could be determined for a catastrophic failure.

APPENDIX G

PHYSICAL PROPERTIES OF UNEXPOSED CRYSTALLINE POLYMERIC MEMBRANES[a] TESTED AT TWO INCHES PER MINUTE

Polymer[b]		HDPE	HDPE[c]	LDPE[c]	LDPE[c]	Polypropylene[c]
Nominal thickness, mil		100	32	10	30	33
Matrecon liner serial number		99	105	21	108	106
	Direction of test					
Physical properties						
Tensile at yield, psi	Machine	2385	3695	1205	1270	4960
	Transverse	2460	4020	1015	1255	4785
Tensile at yield, ppi	Machine	265	111	11.1	37.7	162
	Transverse	244	121	9.7	36.9	159
Elongation at yield, %	Machine	15	17	28	20	7
	Transverse	15	22	18	18	6
Tensile at break, psi	Machine	3915	4270	2845	1690	5800
	Transverse	4440	3295	2645	1645	4570
Tensile at break, ppi	Machine	437	129	26.1	50.2	190
	Transverse	441	98.8	25.2	48.2	152
Elongation at break, %	Machine	925	825	490	515	665
	Transverse	1015	860	635	535	640
Tensile set, %	Machine	819	715	365	400	560
	Transverse	900	715	515	430	545
S-100, psi	Machine	1660	2725	1445	1230	3255
	Transverse	1720	2440	1105	1190	2820
S-100, ppi	Machine	184	82.1	13.2	36.6	106
	Transverse	170	73.2	10.5	34.9	93.9
S-200, psi	Machine	1655	2725	1610	1185	3400
	Transverse	1720	2460	1125	1145	3080
S-200, ppi	Machine	184	82.1	14.8	35.2	111
	Transverse	170	73.9	10.7	33.6	103
Tear strength (Die C), lb	Machine	87.2	32.4	3.9	14.2	35.3
	Transverse	88.9	28.1	3.7	12.6	31.7
Tear strength (Die C), ppi	Machine	839	992	424	462	1082
	Transverse	850	897	400	421	987

[a] Tensile properties measured on 0.25 in. wide "special" dumbbell tested at 2 inches per minute in accordance with ASTM D638. Tear resistance measured in accordance with ASTM D1004 at a test speed of 2 in. per minute.

[b] HDPE = High-density polyethylene; LDPE = low-density polyethylene.

[c] Compounded without pigment.

APPENDIX H

MATERIALS USED IN CONSTRUCTING PRIMARY EXPOSURE CELLS AND MOUNTING LINER SPECIMENS

Material	Use	Trade name	Supplier
Epoxy resins			
Epoxy 1	To cast epoxy rings for sealing liners in cells[a]	Concresive 1217	Adhesive Engineering
Epoxy 2	To coat inner walls of cells and bases; to cast sealing rings[a]	Concresive 1305	Adhesive Engineering
Epoxy 3	To cast epoxy rings[a]	Concresive 1310	Adhesive Engineering
Epoxy 4	To seal 2-in. cylindrical specimens in glass cylinders	Concresive 1001	Adhesive Engineering
Paints			
Primer	To coat inner surface of cells before coating with epoxy	Koropon CA	De Soto, Berkeley, CA
Caulks			
Butyl rubber	To seal membrane specimens in cells	Sure-Seal	Carlisle, Carlisle, PA
Polysulfide	To seal membrane specimens in cells	Colma Joint Sealer (Sikaflex 412)	Sika, Lyndhurst, NJ
Gasket			
Neoprene sponge	To seal liners in primary exposure cells	...	Cal Neva, Oakland, CA
Silica gravel	Inert gravel for base of cells.	...	G.E. Dodson Auburn, CA
Sandblasting sand	To cast epoxy rings to use on inside surfaces of spacers	Clementina No. 2	Clementina, Berkeley, CA

[a] The first rings were made with Epoxy 1; however, as Epoxy 2 and 3 are more resistant to chemicals and solvents, we decided to use them in the remainder of the cells.

APPENDIX I

PHYSICAL PROPERTIES OF PRIMARY LINERS AFTER WASTE EXPOSURE IN CELLS

Polymer	Liner no.	Exposure: Waste[a]	Exposure: Time, days	Thickness, mil	Analytical properties: Volatiles, %	Analytical properties: Extractables, %	Physical properties: Tensile strength[b]	Physical properties: Elongation[b]	Physical properties: S-100[b]	Physical properties: S-200[b]	Physical properties: Tear strength[b]	Puncture resistance: Stress	Puncture resistance: Elongation	Hardness: Duro A	Hardness: Duro D
Butyl	57R		Unexp.	34	0.29	6.36	72.7 ppi[c]	42%[d]	...	...	...	26.6 lb	0.26 in.	71A	...
							Retention of properties, %							Change in points	
		HNO_3-HF-HOAc (W-9)	505	34	5.92	...	96	60	...	...	...	85	92	-4A	...
			1218	37	11.46	8.65	94	645	...	...	...	85	100	-5A	...
		Pesticide (W-11)	500	35	4.10	5.15	111	143	...	...	...	97	123	-4A	...
			1258	34	4.79	7.62	93	100	...	...	...	105	131	+1A	...
		Pb waste (W-14)	499	35	2.79	7.75	97	119	...	...	...	91	108	-4A	...
			1339	34	3.53	7.86	98	167	...	...	...	94	119	-8A	...
		Spent caustic (W-2)	526	34	1.75	...	104	60	...	...	...	104	92	-2A	...
			1249	34	1.37	7.86	100	219	...	...	...	113	131	-2A	...

Continued

APPENDIX I. (Continued)

Polymer	Liner no.	Exposure		Thickness, mil	Analytical properties		Physical properties								
		Waste[a]	Time, days		Volatiles, %	Extractables, %	Tensile strength[b]	Elongation[b]	S-100[b]	S-200[b]	Tear strength[b]	Puncture resistance		Hardness	
												Stress	Elongation	Duro A	Duro D
Chlorinated polyethylene	77		Unexp.	29	0.14	9.13	2197 psi	402%	900 psi	1180 psi	7.38 lb	43.9 lb	0.94 in.	80A	...
							Retention of properties, %							Change in points	
		HNO$_3$-HF-HOAc (W-9)	459	32	7.82	10.09	93	89	97	102	82	95	99	-8A	...
			1218	35	13.18	9.41	101	89	113	118	75	108	101	-13A	...
		Oil Pond 104 (W-5)	521	36	3.69	...	55	98	53	58	55	70	101	-30A	...
			1358	38	10.11	17.00	58	88	62	68	63	88	133	-16A	...
		Pesticide (W-11)	500	31	4.99	9.72	96	100	94	98	99	109	112	-3A	...
			1258	32	7.91	9.41	101	89	113	118	104	108	101	-6A	...
		Pb waste (W-14)	499	36	11.58	7.31	68	101	56	60	77	79	98	-13A	...
			1344	40	19.20	7.24	66	83	71	76	83	77	99	-21A	...
		Spent caustic (W-2)	526	28	2.32	...	93	107	87	84	84	102	120	-5A	...
			1249	29	2.79	9.10	114	88	129	131	100	95	85	-3A	...
	86		Unexp.	22	0.05	...	1677 psi	475%	572 psi	807 psi	3.98 ppi	20.9 lb	0.91 in.	67A	...
							Retention of properties, %							Change in points	
		50% Well 118/ 50% H$_2$O	985	26	16.50	10.77	83	81	84	90	107	132	104	-5A	...

Continued

APPENDIX I. (Continued)

		Exposure			Analytical properties		Physical properties								
												Puncture resistance		Hardness	
Polymer	Liner no.	Waste[a]	Time, days	Thick-ness, mil	Vola-tiles, %	Extract-ables, %	Tensile strength[b]	Elonga-tion[b]	S-100[b]	S-200[b]	Tear strength[b]	Stress	Elong-ation	Duro A	Duro D
Chlorosul-fonated polyethy-lene	6R		Unexp.	31	0.51	3.77	1727 psi	242%	937 psi	1550 psi	...	33.7 lb	0.59 in.	77A	...
							Retention of properties, %							Change in points	
		HNO_3-HF-HOAc (W-9)	505	35	4.69	....	116	90	10>	106	...	135	112	-3A	...
			1218	35	7.18	4.62	134	79	112	74	...	136	115	-4A	...
		Oil Pond 104 (W-5)	521	41	7.51	...	130	103	63	96	...	162	147	-32A	...
			1357	42	10.25	9.45	108	72	96	...	...	177	139	-17A	...
		Pesticide (W-11)	504	35	8.00	4.13	120	112	90	109	...	125	132	-4A	...
			1258	33	9.73	5.39	137	85	118	149	...	162	141	-4A	...
		Pb waste (W-14)	499	36	10.78	3.52	116	107	85	104	...	153	120	-11A	...
			1343	37	11.44	5.95	134	77	118	78	...	151	119	-13A	...
		Spent caustic (W-2)	526	33	4.77	...	153	70	165	...	...	154	90	-2A	...
			1249	34	5.77	3.79	167	65	200	...	...	180	114	+2A	...
	125R[e]		Unexp.	33	0.12	8.97	49.8 ppi	233%	35.4 ppi	47.2 ppi	...	30.6 lb	0.61 in.	75A	28D
							Retention of properties, %							Change in points	
		D.I. Water (W-18)	985	37	14.45	7.77	107	113	89	101	...	128	75	-11A	-2D
		Well 118 (W-17)	990	38	9.03	8.70	116	101	96	114	...	138	128	-5A	-13D

Continued

APPENDIX I. (Continued)

Polymer	Liner no.	Exposure		Thickness, mil	Analytical properties		Physical properties								
		Waste[a]	Time, days		Volatiles, %	Extractables, %	Tensile strength[b]	Elongation[b]	S-100[b]	S-200[b]	Tear strength[b]	Puncture resistance Stress	Puncture resistance Elongation	Hardness Duro A	Hardness Duro D
Elasticized polyolefin	36		Unexp.	23	0.15	5.50	2620 psi	665	922 psi	1017 psi	8.56 lb	26.3 lb	0.97 in.	89A	32D
							Retention of properties, %							Change in points	
		Slurry oil (W-15)	327	26	0.38	...	67	96	70	68	76	114	164	-12A	...
			2355	26	4.02	23.88	57	97	60	58	127	70	107	-27A	-11D
		HNO_3-HF-HOAc (W-9)	505	24	3.20	5.40	98	99	92	96	109	152	146	-5A	...
			1217	23	5.26	7.09	98	96	98	99	100	135	128	-3A	...
		Oil Pond 104 (W-5)	521	26	2.15	...	44	86	61	59	56	85	120	-18A	...
			1357	27	5.12	20.74	52	78	71	69	76	86	124	-16A	...
		Pesticide (W-11)	494	24	0.13	7.14	102	101	104	107	103	130	130	-1A	...
			2699	23	0.58	6.86	96	97	91	91	89	103	113	...	0D
		Pb waste (W-14)	499	24	1.03	5.66	72	92	95	92	96	113	119	-2A	...
			1343	23	1.53	8.06	86	94	104	102	113	118	119	-9A	-1D
		Spent caustic (W-2)	526	23	1.25	...	90	100	87	88	103	111	111	-4A	...
			2677	24	1.01	5.96	94	97	87	88	92	98	110	...	+1D
		Well 118 (W-17)	990	24	1.17	4.90	94	96	103	100	110	127	137	-3A	+2D
		50% Well 118/ 50% H_2O (W-21)	985	24	1.23	5.48	98	98	103	100	107	111	104	-1A	0D
		Deionized H_2O (W-18)	981	21	0.57	5.19	101	96	104	102	93	106	94	-8A	0D
		HFL (W-10)	2293	23	1.46	5.40	100	98	98	98	98	110	118	...	0D
		Slop water (W-4)	2299	25	...	...	80	88	97	95	91	145	140	-16A	-2D

Continued

APPENDIX I. (Continued)

		Exposure			Analytical properties		Physical properties								
												Puncture resistance		Hardness	
Polymer	Liner no.	Waste[a]	Time, days	Thick-ness, mil	Vola-tiles, %	Extract-ables, %	Tensile strength[b]	Elonga-tion[b]	S-100[b]	S-200[b]	Tear strength[b]	Stress	Elong-ation	Duro A	Duro D
Ethylene propylene rubber	26		Unexp.	36	0.50	22.96	1900 psi	450%	357 psi	877 psi	7.40 lb	31.3 lb	1.24 in.	58A	...
							Retention of properties, %							Change in points	
		HNO$_3$-HF-HOAc (W-9)	497	37	8.95	21.36	91	97	81	93	88	118	106	-8A	...
			1147	38	12.02	27.76	79	94	70	81	87	105	100	-12A	...
		Pesticide (W-11)	500	39	3.34	23.13	96	100	89	96	103	110	110	-5A	...
			1258	38	6.29	25.20	96	104	87	67	98	104	108	-6A	...
		Pb waste (W-14)	499	38	2.83	22.27	92	100	84	92	86	94	93	-1A	...
			1344	39	5.25	26.01	94	106	80	92	101	104	113	-8A	...
		Spent caustic (W-2)	526	33	1.27	...	104	102	88	86	90	103	99	-2A	...
			1249	38	1.31	23.95	102	95	108	112	103	118	111	-2A	...

Continued

APPENDIX I. (Continued)

Polymer	Liner no.	Exposure: Waste[a]	Exposure: Time, days	Thickness, mil	Analytical properties: Volatiles, %	Analytical properties: Extractables, %	Tensile strength[b]	Elongation[b]	S-100[b]	S-200[b]	Tear strength[b]	Puncture resistance: Stress	Puncture resistance: Elongation	Hardness: Duro A	Hardness: Duro D
Neoprene	43		Unexp.	34	0.45	13.69	1758 psi	320%	460 psi	1037 psi	5.41 lb	30.6 lb	1.14 in.	57A	...
							Retention of properties, %							Change in points	
		Basin F (W-16)	136	38	11.92	...	84	88	83	94	80	112	89	-1A	...
			1386	37	9.11	13.50	60	70	105	95	75	85	72	0A	...
		Oil Pond 104 (W-5)	521	41	12.99	...	59	86	50	67	43	85	103	-20A	...
			1356	38	21.31	15.86	62	92	42	51	39	72	121	-23A	...
		Pesticide (W-11)	494	37	11.29	13.25	80	93	62	79	65	89	99	-9A	...
			1257	38	13.63	16.14	61	83	54	70	59	113	132	-17A	...
		Pb waste (W-14)	499	42	18.01	8.95	52	76	53	71	47	95	99	-17A	...
			1342	43	17.50	12.15	56	75	61	75	58	98	110	-19A	...
		Spent caustic (W-2)	526	33	4.40	...	98	98	90	98	78	97	92	+1A	...
			1237	35	5.67	13.69	100	95	95	105	97	105	104	+3A	...
		Well 118 (W-17)	136	41	15.53	...	70	82	65	81	62	105	89	-10A	...
			1368	42	25.88	12.11	52	69	66	78	59	87	85	-8A	...

Continued

APPENDIX I. (Continued)

		Exposure			Analytical properties		Physical properties								
												Puncture resistance		Hardness	
Polymer	Liner no.	Waste^a	Time, days	Thickness, mil	Volatiles, %	Extractables, %	Tensile strength^b	Elongation^b	S-100^b	S-200^b	Tear strength^b	Stress	Elongation	Duro A	Duro D
Polyester elastomer	75		Unexp.	7	0.26	2.74	6767 psi	575%	2585 psi	2732 psi	5.92 lb	29.9 lb	1.30 in.	93A	45D
							Retention of properties, %							Change in points	
		Slurry oil (W-15)	328	7	0.40	...	71	77	77	85	76	66	82	-10A	...
		HNO_3-HF-HOAc (W-9)	323	7	0.39	10.77	33	0.3	...	...	24	8	14	-15A	...
			509	8	4.74	13.36	8	4	...	...	5	2	15	-23A	...
		Oil Pond 104 (W-5)	521	8	1.27	...	115	95	94	100	88	95	86	-10A	...
			1357	9	2.59	7.28	89	92	85	88	105	57	63	-21A	...
		Pesticide (W-11)	501	8	0.60	5.15	84	96	95	97	109	90	104	-1A	...
			1258	8	2.92	5.83	72	87	96	94	109	70	74	-15A	...
		Pb waste (W-14)	499	7	2.63	2.98	88	98	88	87	89	69	87	-3A	...
			1342	10	1.72	5.35	70	90	88	87	127	75	49	-12A	+4D
		Spent caustic (W-2)	526	6	0.65	...	86	86	101	101	86	81	93	-2A	...
			1237	7	0.89	3.31	88	86	109	106	107	80	76	-8A	...

Continued

APPENDIX I. (Continued)

Polymer	Liner no.	Exposure: Waste[a]	Exposure: Time, days	Thickness, mil	Analytical properties: Volatiles, %	Analytical properties: Extractables, %	Physical properties: Tensile strength[b]	Elongation[b]	S-100[b]	S-200[b]	Tear strength[b]	Puncture resistance: Stress	Puncture resistance: Elongation	Hardness: Duro A	Hardness: Duro D
Polyvinyl chloride	59		Unexp.	33	0.31	35.86	2557 psi	375%	995 psi	1580 psi	9.89 lb	40.0 lb	0.75 in.	73A	26D
							Retention of properties, %							Change in points	
		Slurry oil (W-15)	327	33	0.29	...	106	90	113	111	106	112	113	-5A	...
		HFL waste (W-10)	1565	32	9.90	34.42	101	88	153	126	123	128	92	+16A	+13D
		HNO_3-HF-HOAc (W-9)	505	33	12.08	16.68	109	82	200	147	172	122	65	+11A	...
			1352	32	13.94	18.58	112	71	249	168	176	126	59	+19A	+28D
		Oil Pond 104 (W-5)	521	32	1.70	32.62	113	85	152	138	126	129	93	+3A	...
			1356	32	4.19	29.99	116	85	174	145	137	150	104	+7A	+9D
		Pesticide (W-11)	500	32	2.30	35.27	101	101	100	96	94	126	137	-3A	...
			1258	32	3.61	33.39	109	95	137	121	114	122	108	+2A	...
		Pb waste (W-14)	499	33	3.34	33.47	86	95	82	82	74	110	113	-4A	...
			1345	34	4.43	22.47	93	93	103	97	110	120	125	-6A	...
		Slop water (W-4)	1565	30	18.72	10.40	93	3	...	...	30	29	32	+15A	+27D
		Spent caustic (W-2)	526	33	2.34	34.62	102	102	99	96	93	104	112	-2A	...
			1249	32	1.85	35.61	106	97	115	108	98	110	113	0A	...
	92		Unexp.	20	0.06	32.75	2290 psi	250%	1440 psi	2030 psi	8.08 lb	27.4 lb	0.62 in.	82A	30D
							Retention of properties, %							Change in points	
		D.I. water (W-18)	981	21	5.54	31.77	110	131	90	89	94	114	94	-3A	-3D
		50% Well 118: 50% H_2O (W-21)	985	19	3.96	33.86	133	116	145	128	143	151	98	+6A	+12D

[a] Matrecon waste serial number given in parentheses.

[b] Average of values obtained in machine and transverse directions.

[c] Value reported is tensile strength of fabric since the bulk of lining material's strength is in the nylon fabric and since the butyl coating over the fabric did not to fail catastrophically. Fabric failed at 25% elongation.

[d] Value reported is elongation at ultimate break. Fabric in sheeting retained its original elongation.

[e] Tensile properties determined in accordance with ASTM D751 Method B using 1-in. wide strips for the unexposed sheeting and 0.5-in. wide strips for the exposed.

APPENDIX J-1

EXPOSURE OF LINER SPECIMENS IN IMMERSION TEST - NUMBER OF DAYS OF IMMERSION

Polymeric membrane liner		Wastes[a]												
		Acidic		Alkaline		Brine	Industrial		Oily			Organic trace	Pesticide	Water
Polymer	Number	"HFL" (W-10)	"HNO_3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste"[b] (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	Sat'd[c] TBP (W-20)	"Weed killer" (W-11)	Deionized (W-18)
Butyl rubber	44	250 761	193 751	193 823	236 780	174 1456	... 1196	238 786	257 761	248 752	252 809	522 1106	242 807	174 1434
Chlorinated polyethylene	77	250 761	193 751	193 823	236 780	174 1456	... 1196	238 786	257 761	248 752	252 ...	522 1112	242 807	174 1434
	100	99 931	267 1253	99 931	99 1258	49 1345	67 1288	97 1257	99 784	97 1252	99 1279	522 1112	97 1259	49 1323
Chlorosulfonated polyethylene	6R	250 761	193 751	193 823	238 780	174 1458	... 1195	236 786	257 761	248 752	252 809	522 1119	242 807	174 1458
	55	250 761	193 751	193 823	236 780	174 1456	... 1196	238 786	257 761	248 752	252 809	522 1119	242 807	174 1434
Elasticized polyolefin	36	250 761	193 751	193 823	238 780	174 1456	... 1196	236 786	257 761	248 752	252 809	522 1090	242 807	174 1434
Ethylene propylene rubber	83R	250 761	193 751	193 823	236 780	174 1456	... 1195	238 786	257 761	248 752	252 809	522 1076	242 807	174 1456
	91	250 761	193 751	193 823	236 780	174 1456	... 1196	238 786	257 761	248 752	252 809	522 1076	242 807	174 1434
Neoprene	90	250 761	193 751	193 823	236 780	174 1456	... 1196	238 786	257 761	248 752	252 809	522 1106	242 807	174 1434
Polyester elastomer	75	250 761	193 751	193 ...	236 780	174 1456	... 1196	238 786	257 761	248 752	252 809	522 1035	242 807	174 1434
Polyethylene														
High density	105	99 934	99 1262	99 934	99 1267	93 1360	67 1287	97 1266	99 793	97 1260	99 1288	522 1070	97 1268	93 1360
Low density	108	99 927	99 1255	99 927	99 1266	93 1360	67 1287	97 1259	99 786	97 1253	99 1287	522 1055	97 1267	93 1322
Polypropylene	106	99 927	99 1255	99 927	99 1260	51 1322	67 1287	97 1259	99 786	97 1253	99 1287	422 1090	97 1261	51 1322
Polyvinyl chloride	11	250 761	193 751	193 823	238 780	174 1456	... 1196	236 786	257 761	248 752	252 809	522 1035	242 807	174 1434
	59	250 761	193 751	193 823	236 780	174 1456	... 1196	238 786	257 761	248 752	252 809	522 1055	242 807	174 1434
	88	250 761	193 751	193 823	236 780	174 1456	... ...	238 786	257 761	248 752	252 809	522[d] 1070[d]	242 807	174 1434

[a] Matrecon waste serial number shown below identification.

[b] Blend of three waste streams.

[c] Saturated solution of tributyl phosphate (TBP) in deionized water.

[d] Liner Number 89.

APPENDIX J-2

EXPOSURE OF LINER SPECIMENS IN IMMERSION TEST - DIMENSIONAL CHANGES IN MACHINE AND TRANSVERSE DIRECTIONS[a]

Polymeric membrane liner		Wastes[b]												
		Acidic		Alkaline		Brine	Industrial		Oily			Organic trace	Pesticide	Water
Polymer	No.	"HFL" (W-10)	"HNO$_3$-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste"[c] (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	Sat'd[d] TBP (W-20)	"Weed killer" (W-11)	Deionized (W-18)
Butyl rubber	44	0.6x0.5 0.3x0.5	0.7x0.5 0.3x0.6	0.3x0.5 0.2x0.2	0x0 0.3x0	0.2x1.0 0x0.7	... -0.2x-0.2	7.2x9.8 14.4x11.7	5.6x10.2 8.8x10.2	27.1x30.2 28.4x32.7	19.9x22.1 20.0x18.9	1.0x0.8 0.5x1.8	0.6x0.2 0.3x0	0x1.0 0.2x0.7
Chlorinated polyethylene	77	0.6x2.0 0x2.2	0.3x1.7 -0.3x4.4	0x1.0 0.3x0	-0.3x0.2 0.3x0.2	0.2x-0.3 -0.2x-0.5	... 0.2x0	-2.7x30.2 -2.0x45.8	0x11.0 e	0.6x9.8 0.2x12.4	-1.6x35.6 ...	6.7x38.3 3.3x46.0	0.3x4.9 0.2x3.7	1.0x1.0 3.7x0.7
	100	1.1x0.3 1.0x1.3	f 5.6x6.8	-0.2x-0.7 -0.2x-0.3	-0.2x0 0x0	0.5x0.6 -0.6x-0.7	0.2x0 -0.2x0	6.8x10.8 16.3x19.6	2.7x3.0 28.1x35.3	4.1x4.2 7.5x8.1	f 32.4x37.0	12.6x11.8 14.4x12.6	0.2x0.7 1.9x2.3	0.2x0.0 1.7x2.3
Chlorosulfonated polyethylene	6R	1.3x1.7 1.3x1.7	0x1.2 2.0x2.4	1.3x1.5 1.3x1.8	0.3x0.7 1.0x0.7	1.0x1.0 0.7x0.7	... f	16.4x22.0 27.2x40.1	11.8x8.8 22.0x25.9	16.4x15.6 8.8x9.0	38.6x53.1 38.2x101	5.3x7.7 4.6x6.3	2.3x3.2 3.0x3.5	2.0x2.0 3.4x2.9
	55	2.3x2.2 2.6x2.9	2.3x2.0 2.5x2.9	1.3x1.5 1.8x2.2	0.6x1.0 1.0x1.2	1.2x2.0 1.7x1.0	... 1.5x2.0	13.1x26.6 25.6x40.9	15.0x18.0 20.1x33.7	16.7x22.7 14.7x21.5	35.3x62.0 34.0x106	11.6x14.3 9.1x11.4	4.2x4.4 5.2x5.7	3.0x3.0 7.3x6.3
Elasticized polyolefin	36	0.3x0.2 0.3x0	0.7x0.7 1.0x0.7	4.3x4.2 4.6x3.5	0.7x0.5 0.3x0.4	0x1.0 -0.2x0.2	... -0.2x-0.2	5.2x5.4 6.2x5.2	6.5x7.1 7.9x7.8	9.5x9.8 7.5x8.3	11.2x12.8 9.7x11.0	3.2x2.5 2.2x2.0	0.3x0.5 0.3x0.2	0x0 -0.5x-0.3
Ethylene propylene rubber	83R	0.7x1.0 0.8x0.5	2.0x2.2 0.3x0.2	0.7x0.5 0.7x0.7	-0.3x0.2 0.7x0.2	0.5x0 -0.2x-0.3	... f	2.0x5.6 3.0x7.1	1.6x2.9 3.3x3.9	3.9x7.3 3.0x4.6	9.2x10.3 11.5x19.8	0.7x2.0 0.7x1.8	0.3x1.2 0.3x1.4	0x0 0.7x1.3
	91	4.0x4.4 5.1x5.8	4.3x5.4 11.5x13.9	1.0x1.2 1.3x1.0	0.3x0 0.7x0.2	1.2x1.0 -0.1x0.9	... -0.2x0.3	10.2x11.0 12.6x13.9	8.8x10.5 8.6x9.8	23.4x27.3 23.3x28.0	21.7x25.1 21.0x23.2	1.4x1.8 2.2x2.0	2.0x2.4 4.9x5.6	1.0x1.0 1.0x0.7
Neoprene	90	2.0x3.4 2.3x3.4	2.6x3.4 4.2x5.3	0.3x0.2 4.0x1.0	0.7x0.5 0.3x0.8	1.0x1.0 -0.3x-1.4	... 0.5x1.0	15.8x26.9 20.8x22.1	17.1x20.4 35.7x39.5	11.3x10.9 8.6x10.7	30.9x32.2 29.9x29.0	15.5x18.7 13.3x15.2	3.0x3.2 3.4x4.2	2.0x2.0 3.7x2.9
Polyester elastomer	75	0.3x0.7 0.3x1.0	1.0x1.2 pieces[g]	0.6x0.7 ...	0.7x0 0.7x0.5	0.7x0.6 0.4x0.8	... 0.2x0	2.0x4.4 3.1x2.4	5.6x5.4 6.6x6.1	2.6x2.7 3.0x2.4	6.2x6.4 5.6x5.4	2.2x2.6 1.0x2.9	0.6x1.2 0.7x0.5	0x1.0 0x0.3
Polyethylene														
High density	105	-0.5x-0.6 0.5x-0.6	-0.2x-0.6 0x0	-0.2x0 2.0x0	-0.2x-0.7 -0.2x0	0x-1.0 0x0	-0.1x-0.4 0.2x0	1.5x0.6 2.5x1.6	0.5x-0.3 3.0x2.0	3.4x2.6 2.7x1.6	1.9x0.9 3.0x1.6	0.8x0.6 0.7x0.2	-0.5x-0.3 -0.5x0.7	0x0 0.7x-0.3
Low density	108	0x-0.7 -0.5x0	-0.2x-0.3 0x0	-0.2x-0.7 0x0.7	-0.5x-0.3 0x0	0x1.0 0.2x-0.3	-0.1x-1.1 0x-0.3	2.0x1.6 4.0x3.3	2.7x2.0 4.7x3.9	1.0x1.3 3.1x3.2	3.9x3.3 5.9x4.2	0.5x0 0.2x0.2	1.9x-0.6 0.3x0.3	0.2x0.3 0.2x0.3
Polypropylene	106	-0.2x-0.3 -0.3x0	0x0 0x0	-0.2x-0.3 0.3x0	-0.2x-0.3 0x0	1.0x0 0.5x0	0.7x-0.4 -0.2x0.6	2.4x0.9 4.5x2.6	-0.2x-1.2 -0.8x0.3	-0.2x-0.7 3.3x1.9	3.6x3.3 4.0x2.0	1.0x-0.3 0.1x0.5	0x-0.3 0.3x-0.3	0x0 0x0
Polyvinyl chloride	11	3.3x3.9 4.9x7.1	5.9x6.6 5.6x7.6	-5.9x-4.4 -4.3x-3.7	0x0 0.3x0	-2.0x-2.0 -2.5x-2.4	... 0.2x0.3	1.6x1.2 -2.6x-0.7	2.6x3.9 2.6x4.8	-4.9x-4.8 -5.6x-5.8	2.6x4.2 4.6x5.4	17.2x22.3 15.9x19.9	1.3x1.2 1.6x1.5	0x-1.0 -1.0x1.3
	59	1.0x1.5 -0.3x0	-1.3x-1.0 -4.3x-3.4	-4.3x-3.4 -6.6x-5.8	0.3x0.2 0x0.2	-2.0x-2.0 -3.3x-3.4	... -0.2x6.8	2.0x3.4 2.4x5.6	2.6x2.4 4.6x6.6	-0.7x-0.2 -1.3x-0.7	10.2x12.4 6.4x10.7	11.3x16.6 10.3x17.3	0x0.5 -0.6x-0.7	0x0 -0.2x-1.6
	88	1.3x2.0 2.0x3.4	3.0x5.2 5.3x7.4	-4.9x-2.2 -4.3x-4.2	0x0 0.3x0.2	-0.5x-0.3 -1.1x-1.3	... ...	1.3x0.7 -4.4x-0.6	0.7x2.0 -0.3x3.7	-4.0x-4.2 -4.6x-4.6	2.6x7.6 5.3x10.3	10.6x20.0[h] 11.3x19.4[h]	0.3x1.2 0x0.5	-0.2x0.0 -0.2x-1.0

[a] Reported values are percent change in machine and transverse directions, respectively. Immersion times for the respective data are presented in Appendix J-1.

[b] Matrecon waste serial number shown below identification.

[c] Blend of three wastes.

[d] Saturated solution of tributyl phosphate (TBP) in deionized water.

[e] Not measured due to condition of immersed specimen which had become very tacky and seemed to have partially dissolved.

[f] Not measured.

[g] Immersed specimen became brittle and broke in pieces when removed from cell.

[h] Liner Number 89.

APPENDIX J-3
EXPOSURE OF LINER SPECIMENS IN IMMERSION TEST - PERCENT INCREASE IN WEIGHT

Polymeric membrane liner		Percent increase in weight of samples on immersion in different wastes[a]												
		Acidic		Alkaline		Brine	Industrial		Oily			Organic trace	Pesticide	Water
Polymer	Number	"HFL" (W-10)	"HNO_3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste"[b] (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	Sat'd[c] TBP (W-20)	"Weed killer" (W-11)	Deionized (W-18)
Butyl rubber	44	2.74 3.71	1.39 3.77	2.04 1.81	0.37 0.74	0.87 1.4	... 1.0	20.1 28.7	32.3 31.18	97.5 104	70.8 64.2	18.1 23.1	0.76 1.57	1.34 4.4
Chlorinated polyethylene	77	9.43 12.9	9.31 19.9	1.50 1.89	0.64 1.11	2.54 1.3	... 2.0	70.9 119	59.5 d	31.6 36.9	117 ...	117 121	9.62 12.7	5.66 12.4
	100	4.2 9.24	2.0 21.2	-0.5 1.83	0.7 0.2	0.85 -1.2	0.3 1.2	23.0 29.3	11.9 115	12.4 20.9	e 118	36.2 37.5	2.8 7.3	1.45 7.5
Chlorosulfonated polyethylene	6R	6.75 8.95	10.3 10.0	3.79 7.65	3.32 4.30	5.75 4.06	... e	83.0 121	51.1 105	75.10 49.5	202 368	31.5 30.1	13.07 17.26	7.99 15.8
	55	5.41 7.74	7.46 10.9	3.84 5.66	2.17 3.28	5.06 5.6	... 5.7	69.6 116	53.2 111	58.5 55.0	211 348	38.3 31.7	12.3 15.7	7.73 18.9
Elasticized polyolefin	36	0.25 1.05	2.68 7.57	17.3 20.7	0.54 0.56	0.09 0.3	... 1.0	18.2 17.0	21.8 29.4	33.5 28.9	44.2 38.1	7.9 9.7	0.00 0.49	-0.04 0.6
Ethylene propylene rubber	83R	3.06 3.05	2.64 4.20	2.71 3.98	1.34 1.59	2.19 1.0	... e	23.0 24.8	15.8 19.8	35.4 26.5	73.4 84.4	6.8 9.8	3.71 4.51	2.62 3.3
	91	16.7 23.9	18.3 50.9	3.13 3.34	0.23 1.30	1.19 1.0	... 1.0	29.3 34.7	35.3 34.2	80.1 84.7	79.4 76.2	5.2 5.9	8.09 20.4	1.93 3.6
Neoprene	90	9.60 12.0	10.8 17.4	0.38 2.66	0.82 1.53	3.54 ...	... 3.2	45.6 59.1	60.7 142.6	25.8 26.3	94.8 89.3	49.4 41.1	8.54 11.4	7.10 11.4
Polyester elastomer	75	0.55 2.03	4.15 6.41	e e	0.64 1.29	-0.69 1.5	... 1.2	7.57 7.40	17.1 16.6	7.90 8.47	16.3 14.7	4.7 4.6	2.39 4.15	0.00 -0.4
Polyethylene														
High density	105	0.05 0.16	0.1 0.2	0.2 0.52	0.2 0.01	0.03 0.09	-0.3 0.1	5.0 4.5	4.4 8.0	3.3 6.6	6.4 7.3	0.33 0.5	0.5 0.2	0.03 0.6
Low density	108	-3.3 0.08	-0.3 0.3	3.5 1.07	0.1 0.1	0.07 0.09	1.1 0.2	3.1 5.3	8.5 12.0	8.4 10.3	10.7 14.0	0.44 0.5	0.2 0.2	0.03 0.2
Polypropylene	106	0.05 -0.02	0.1 -0.01	0.1 0.08	0.2 0.1	-0.11 -0.05	0.7 0.5	6.9 5.9	0.4 1.4	0.6 6.8	1.4 9.1	0.33 -1.3	0.07 -0.1	-0.01 0.00
Polyvinyl chloride	11	10.2 18.1	16.8 22.1	-13.5 -11.1	0.09 0.43	-4.81 -6.2	... 1.0	4.36 -1.54	10.7 18.5	-7.65 -10.4	10.0 14.3	57.7 52.8	4.03 5.13	0.21 -1.6
	59	2.76 0.86	-2.82 -6.12	-6.35 -15.7	-3.00 -0.89	-4.82 -7.8	... 0.7	8.81 7.39	11.3 28.9	-1.54 -0.54	33.4 24.7	39.7 40.7	0.46 0.95	1.18 -0.5
	88	7.60 14.3	19.8 28.2	-13.5 -12.1	0.04 1.08	-1.51 -1.8	... ...	2.22 -5.15	7.2 14.1	-10.3 -9.9	18.1 25.2	47.6[f] 47.5[f]	2.89 1.62	0.65 -0.1

[a] Matrecon waste serial number shown below identification. Immersion times for the respective data are presented in Appendix J-1.

[b] Blend of three waste streams.

[c] Saturated solution of tributyl phosphate (TBP) in deionized water.

[d] Not measured because immersed specimen had become very "gooey" and seemed partially dissolved.

[e] Not measured.

[f] Liner Number 89.

APPENDIX J-4
EXPOSURE OF LINER SPECIMENS IN IMMERSION TEST - PERCENT VOLATILES AFTER IMMERSION

Polymeric membrane liner			Wastes[a]												
			Acidic		Alkaline		Brine	Industrial		Oily			Organic trace	Pesticide	Water
Polymer	No.	Original values	"HFL" (W-10)	"HNO$_3$-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste"[b] (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	Sat'd[c] TBP (W-20)	"Weed killer" (W-11)	Deionized (W-18)
Butyl rubber	44	0.46	1.91 2.95	2.41 3.85	1.32 1.24	0.67 0.71	d 2.40	... 1.60	3.67 15.34	1.13 3.29	2.99 9.78	14.47 19.14	2.68 5.20	1.14 3.04	d 4.30
Chlorinated polyethylene	77	0.14	6.87 8.47	12.52 20.42	2.36 5.50	1.46 1.60	d 3.30	... 3.20	23.62 42.24	0.59 [e]	6.90 5.22	23.04 ...	42.29 56.60	7.77 8.63	d 11.30
	100	0.66	5.50 7.40	d 18.70	2.96 6.91	2.42 2.63	d 2.80	d 2.80	15.01 18.20	3.77 2.90	9.56 13.00	63.15 57.80	20.33 29.00	3.79 9.37	d 9.30
Chlorosulfonated polyethylene	6R	0.51	4.79 8.01	2.61 10.42	7.46 6.53	3.67 4.20	d 6.20	... 6.40	17.86 33.01	0.54 6.05	10.79 20.22	31.52 50.74	20.51 21.60	9.00 10.79	d 13.20
	55	0.42	4.98 6.91	8.54 9.14	1.98 5.96	2.73 3.62	d 6.40	... 6.50	13.09 24.40	0.72 4.82	11.07 9.98	18.71 39.71	21.13 23.10	8.75 7.94	d 15.80
Elasticized polyolefin	36	0.15	0.70 1.93	3.19 7.21	10.88 11.37	0.72 0.58	d 0.60	... 0.90	2.04 9.46	0.54 1.50	1.80 3.07	14.24 16.37	2.22 6.50	0.28 0.45	d 0.50
Ethylene propylene rubber	83R	0.31	2.49 3.22	6.67 3.09	3.47 4.41	1.43 2.16	d 3.20	... 3.80	4.59 13.89	0.96 4.36	4.06 8.54	6.29 24.09	6.11 8.40	2.99 4.89	d 5.50
	91	0.34	15.52 19.59	16.23 22.55	2.95 2.97	0.90 1.22	d 2.50	... 2.60	6.48 18.47	0.86 4.20	18.35 11.76	30.88 33.37	4.65 6.60	6.17 15.61	d 4.00
Neoprene	90	0.37	8.71 10.88	12.27 13.67	8.96 9.70	2.90 2.93	d 4.90	... 5.20	14.48 23.63	0.74 2.90	9.03 6.26	35.39 28.89	25.14 29.70	7.04 7.26	d 12.40
Polyester elastomer	75	0.26	0.12 2.11	1.36 2.83	2.06 ...	0.39 1.27	d 0.60	... 1.30	1.15 5.77	0.60 3.40	1.46 2.11	9.98 9.48	4.38 5.60	0.48 1.75	d 0.40
Polyethylene															
High density	105	0.14	0.02 0.21	0.34 d	0.19 0.36	0.09 d	d 0.01	d d	1.76 d	1.00 0.60	3.74 d	4.60 d	d 0.50	0.12 d	d 1.10
Low density	108	0.18	0.03 0.25	0.04 d	0.41 0.42	0.03 d	d 0.10	d d	1.09 d	1.67 0.80	2.07 d	4.84 d	d 0.50	0.14 d	d 0.07
Polypropylene	106	0.01	0.12 0.36	0.35 d	0.11 0.28	0.11 d	d 0.20	d 0.07	3.27 d	0.25 0.50	0.40 d	0.83 d	0.01 0.30	0.15 d	d 0.24
Polyvinyl chloride	11	0.15	10.43 17.30	16.20 22.61	11.03 13.36	0.44 0.56	d 2.10	... 4.00	4.74 16.14	0.53 3.62	2.19 2.69	17.07 17.14	14.80 35.10	1.59 3.09	d 2.50
	59	0.31	3.89 3.00	11.68 10.58	2.41 7.84	1.06 1.49	d 2.30	... 0.04	2.93 12.73	0.56 4.56	1.47 3.26	20.98 16.13	12.12 30.60	2.58 4.01	d 3.30
	88	0.17	7.38 13.32	21.39 23.18	16.96 13.59	0.89 1.21	d 2.40	... ...	3.61 11.53	0.59 3.50	2.11 4.33	23.34 24.07	23.33[f] 24.30[f]	2.61 3.53	d 2.90

[a] Matrecon waste serial number shown below identification. Immersion times for the respective data are presented in Appendix J-1.

[b] Blend of three waste streams.

[c] Saturated solution of tributyl phosphate (TBP) in deionized water.

[d] Not measured.

[e] Not measured because of condition of specimen.

[f] Liner Number 89. Original value = 0.03%.

APPENDIX J-5

EXPOSURE OF LINER SPECIMENS IN IMMERSION TEST - PERCENT EXTRACTABLES AFTER IMMERSION

Polymeric membrane liner			Wastes^a												
			Acidic		Alkaline		Brine	Industrial		Oily			Organic trace	Pesticide	Water
Polymer	No.	Original values	"HFL" (W-10)	"HNO_3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste"^{b} (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	Sat'd^c TBP (W-20)	"Weed killer" (W-11)	Deionized (W-18)
Butyl rubber	44	11.79	... 10.62	... 11.07	... 12.81	... 10.87	... ...	... ...	... 17.08	... 27.23	31.17 40.34	... 15.16	12.26 11.89	... 11.03	... ...
Chlorinated polyethylene	77	9.13	... 10.19	... 12.04	... 6.45	... 9.18	... ...	... ...	... 16.62	... ...	16.57 19.18	... ...	5.71 26.1	... 9.07	... ...
	100	17.42	... 16.70	... 18.70	... 15.37	... 18.01	... ...	... ...	... ...	... 57.48	... 16.09	... 36.48	15.24 16.82	... 15.86	... ...
Chlorosulfonated polyethylene	68	3.77	... 3.77	... 3.75	... 3.63	... 3.83	... ...	... ...	... 4.56	... 45.43	25.93 16.34	... 19.01	3.60 3.67	... 3.68	... ...
	55	4.08	... 3.64	... 3.96	... 3.70	... 4.16	... ...	... ...	... 3.74	... 59.81	17.02 15.92	... 16.08	3.97 4.35	... 4.50	... ...
Elasticized polyolefin	36	5.50	... 5.07	... 6.22	... 2.96	... 5.46	... ...	... ...	... 6.86	... 23.31	18.60 17.78	... 8.08	6.29 6.70	... 6.00	... ...
Ethylene propylene rubber	83B	18.16	... 17.12	... 17.63	... 18.17	... 18.02	... ...	... ...	... 20.74	... 27.34	19.60 22.24	... 18.65	16.74 18.81	... 17.69	... ...
	91	23.64	... 22.43	... 25.36	... 24.70	... 22.89	... ...	... ...	... 29.99	... 38.35	31.63 43.45	... 25.43	22.11 23.41	... 24.71	... ...
Neoprene	90	21.46	... 19.58	... 19.18	... 17.63	... 20.83	... ...	... ...	... 19.21	... 58.47	25.32 23.85	... 7.60	16.85 21.46	... 20.52	... ...
Polyester elastomer	75	2.74	... 3.28	... 10.77	... ...	... 2.71	... ...	... ...	... 3.25	... 16.64	... 6.54	... 6.10	3.25 ...	... 3.39	... ...
Polyethylene															
High density	105	...	... 0.97	... ...	... 0.53	... ...	... ...	... ..	... ...	... 3.85	... 3.18	... 1.80	... 0.81	... ...	... ...
Low density	108	2.07	... 3.77	... ...	... 2.91	... ...	... ...	... ...	... ...	... 8.25	... 2.49	... 4.70	2.67 3.94	... ...	... ...
Polypropylene	106	...	... 0.74	... ...	... 0.69	... ...	... ...	... ...	... ...	... 1.20	... 7.67	... 1.41	... 1.44	... ...	... ...
Polyvinyl chloride	11	33.90	... ...	... 32.25	... 1.34	... 33.89	... ...	... ...	... ...	... 40.55	50.43 17.95	... 23.84	23.78 33.28	... 35.38	... 31.75
	59	35.86	... 33.95	... 23.25	... 14.70	... 35.81	... ...	... ...	... 35.81	... 47.56	55.56 27.95	... 31.85	26.14 51.66	... 35.90	... 35.20
	88	33.46	... ...	... 33.20	... 2.15	... 32.53	... ...	... ...	... ...	... 38.49	20.74 14.81	... 21.52	24.90^d 25.84^d	... 31.71	... ...

[a] Matrecon waste serial number shown below identification. Immersion time for the respective data are presented in Appendix J-1. Extractables determined in accordance with Matrecon Test Method 2 (Matrecon, 1983, pp. 340-43).

[b] Blend of three waste streams.

[c] Saturated solution of tributyl phosphate (TBP) in deionized water.

[d] Liner Number 89. Original value = 25.17%.

APPENDIX J-6
EXPOSURE OF LINER SPECIMENS IN IMMERSION TEST - RETENTION OF STRESS AT 100% ELONGATION

Polymeric membrane liner		Original value[b], psi	Retention of original property on immersion in different wastes[a],%												
			Acidic		Alkaline		Brine	Industrial		Oily			Organic trace	Pesticide	Water
Polymer	Number		"HFL" (W-10)	"HNO3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste"[c] (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	Sat'd[d] TBP (W-20)	"Weed killer" (W-11)	Deionized (W-18)
Butyl rubber	44	308	84 93	70 88	85 89	81 91	103 89	... 76	59 57	66 55	44 45	36-47[e] 38	79 60	78 89	90 89
Chlorinated polyethylene	77	900	98 117	92 129	113 130	115 126	124 152	... 126	37 18	44 [f]	44 48	8 ...	6 9	102 103	129 123
	100	618	93 79	[g] 45	96 110	88 119	91 121	103 85	67 44	74 49	62 77	24 28	48 55	76 136	125 108
Chlorosulfonated polyethylene	6R	938	98 126	81 60	149 180	150 171	115 [g]	... [g]	98 89	90 73	49 88	7-46[e] [f]	[g] [g]	109 124	142 [g]
	55	880	91 110	100 71	119 169	130 164	104 134	... 152	79 91	95 85	58 85	34 [f]	80 106	102 120	143 106
Elasticized polyolefin	36	923	97 105	99 93	82 93	95 109	102 114	... 119	80 76	70 72	62 75	55-59[e] 54	71 87	108 116	123 104
Ethylene propylene rubber	83R	760	92 116	107 88	69 63	90 107	104 [g]	... [g]	60 47	63 61	38 57	28 [f]	[g] [g]	98 100	118 [g]
	91	338	86 100	75 58	88 107	93 100	85 99	... 104	83 73	65 60	66 58	59-64[e] 67	82 89	85 89	109 93
Neoprene	90	558	82 104	83 62	96 115	97 121	106 144	... 93	50 38	58 40	46 70	25-26[e] 27	37 38	84 109	126 109
Polyester elastomer	75	2585	98 117	80 [h]	90 [g]	94 104	109 114	... 103	91 91	79 75	82 95	68-69[e] 77	90 90	99 110	117 105
Polyethylene High density	105	2510 2583[j]	101 89[i]	103 79[i]	[h] 90[i]	101 95[i]	[h] 104[i]	[h] 83[i]	100 82[i]	99 86[i]	102 88[i]	98 83[i]	92[i] 89[i]	100 83[i]	[h] 98[i]
Low density	108	1320 1210[j]	97 101[i]	99 105[i]	97 98[i]	100 99[i]	104 103[i]	100 102[i]	95 93[i]	91 100[i]	92 90[i]	88 89[i]	104[j] 103[i]	94 100[i]	111 105[i]
Polypropylene	106	[k] 3038[j]	[k] 101[i]	[k] 98[i]	[k] 98[i]	[k] 106[i]	[k] 105[i]	[k] 102[i]	[k] 95[i]	[k] 108[i]	[k] 100[i]	[k] 97[i]	109[i] 107[i]	[k] 99[i]	[k] 108[i]
Polyvinyl chloride	11	1420	83 95	84 93	196 206	103 110	116 139	... 95	82 91	89 118	168 186	48-70[e] 46	15 18	99 106	111 108
	59	995	99 124	199 252	161 239	103 123	136 185	... 106	89 87	114 107	118 145	32-35[e] 45	27 28	118 130	113 116
	88	1735	70 83	68 70	183 [m]	86 99	96 125	... ...	83 95	99 122	145 172	37-41[e] 45	23[i] 25[l]	83 99	100 104

[a] Matrecon waste serial number shown below identification. Immersion times for the respective data are given in Appendix J-1.
[b] Average of values in machine and transverse directions of specimens tested in accordance with ASTM D412 or D638 at 20 in. per minute unless otherwise noted.
[c] Blend of three waste streams.
[d] Saturated solution of tributyl phosphate (TBP) in deionized water.
[e] Waste stratified into an aqueous and an oily phase. Reported value ranges indicate the different effects of the two phases.
[f] Not measured because of condition of immersed specimen.
[g] Not measured.
[h] Immersed sample failed at less than 100% elongation.
[i] Retention of values obtained for specimens tested at 2 in. per minute.
[j] Average of values in machine and transverse directions of specimens tested at 2 in. per minute.
[k] Unexposed material failed at less than 100% elongation when tested at 20 in. per minute.
[l] Liner Number 89. Original value = 1688 psi.
[m] Transverse direction failed at 80% elongation.

APPENDIX J-7
EXPOSURE OF LINER SPECIMENS IN IMMERSION TEST - RETENTION OF ELONGATION AT BREAK

Polymeric membrane liner			Retention of original property on immersion in different wastes[a], %												
			Acidic		Alkaline		Brine	Industrial		Oily			Organic trace	Pesticide	Water
Polymer	Number	Original value[b], %	"HFL" (W-10)	"HNO3-HF-HOAc" (W-9)	"Slop water" (W-4)	"Spent caustic" (W-2)	5% NaCl (W-19)	"Basin F" (W-16)	"Lead waste"[c] (W-14)	"Slurry oil" (W-15)	"Oil Pond 104" (W-5)	"Weed oil" (W-7)	Sat'd[d] TBP (W-20)	"Weed killer" (W-11)	Deionized (W-18)
Butyl rubber	44	443	109 93	109 110	103 100	90 93	98 102	... 104	115 111	93 101	52 54	43-100[e] 71	109 109	98 109	100 99
Chlorinated polyethylene	77	403	93 79	87 77	88 83	95 86	65 75	... 77	87 89	97 f	100 93	42 ...	154 139	96 92	77 68
	100	403	87 77	g 92	90 73	92 77	91 87	84 87	94 87	102 58	100 72	51 40	78 78	101 67	81 73
Chlorosulfonated polyethylene	6R	243	106 89	137 121	62 54	89 69	93 g	... g	72 62	81 74	70 76	83 g	g g	97 78	82 g
	55	280	111 93	83 104	71 64	75 73	94 82	... 73	81 60	65 51	79 69	82 f	79 73	101 79	79 87
Elasticized polyolefin	36	665	108 95	102 96	84 100	97 90	100 95	... 93	89 84	96 89	86 88	63-77[e] 78	73 70	101 95	93 97
Ethylene propylene rubber	83R	253	144 109	109 136	147 147	107 105	121 g	... g	134 156	134 146	154 133	124 f	g g	120 126	101 g
	91	488	98 87	104 97	98 93	107 106	103 102	... 100	92 98	92 100	47 52	45-53[e] 56	106 103	109 101	105 100
Neoprene	90	415	98 68	86 87	82 81	98 83	83 76	... 80	97 91	77 62	98 77	47-70[e] 64	82 88	96 86	75 74
Polyester elastomer	75	575	97 90	46 1	86 g	96 83	89 85	... 86	102 101	94 87	98 96	96-99[e] 96	101 96	96 89	83 93
Polyethylene High density	105	113 843[i]	92 102[h]	130 17[h]	86 99[h]	100 44[h]	78 62[h]	74 31[h]	129 82[h]	134 92[h]	196 79[h]	125 87[h]	100[h] 42[h]	113 43[h]	53 91[h]
Low density	108	555 525[i]	99 98[h]	100 86[h]	99 90[h]	103 89[h]	99 96[h]	108 89[h]	105 67[h]	95 93[h]	96 66[h]	86 48[h]	102[h] 82[h]	107 87[h]	101 88[h]
Polypropylene	106	58 653[i]	91 95[h]	140 92[h]	91 98[h]	91 94[h]	132 97[h]	47 76[h]	942 108[h]	82 112[h]	204 104[h]	270 107[h]	104[h] 92[h]	168 94[h]	207 94[h]
Polyvinyl chloride	11	358	91 83	87 84	58 35	102 95	101 87	... 94	101 101	92 74	87 83	99-103[e] 105	103 99	104 101	105 90
	59	375	103 95	82 67	91 57	102 89	100 80	... 90	106 103	92 80	98 98	104-111[e] 98	91 96	101 98	97 89
	88	330	108 87	91 86	33 35	108 100	97 81	... ...	108 104	85 82	78 78	112-114[e] 107	111[j] 105[j]	95 98	102 89

[a] Matrecon waste serial number shown below identification. Immersion times for the respective data are presented in Appendix J-1.

[b] Average of values in machine and transverse directions of specimens tested in accordance with ASTM D412 or D638 at 20 in. per minute unless otherwise noted.

[c] Blend of three waste streams.

[d] Saturated solution of tributyl phosphate (TBP) in deionized water.

[e] Waste stratified into an aqueous phase and an oily phase. Reported value ranges indicate the different effects of the two phases.

[f] Not measured because of condition of specimen.

[g] Not measured.

[h] Retention of values obtained for specimens tested at 2 in. per minute.

[i] Average of values in machine and transverse directions tested at 2 in. per minute.

[j] Liner Number 89. Original value = 320%.

APPENDIX K. (Continued)

Polymer	Chlorosulfonated polyethylene				Elasticized polyolefin			
Compound type[b]	TP				CX			
Fabric, type	Nylon				...			
Thread count, epi	8x8				...			
Nominal thickness, mils	31				22			
Liner number[d]	6R				36			
Exposure time, days	...	343	745	1231	...	343	745	1231
Weight, % change	...	0.64	0.91	1.80	...	-0.72	-1.55	-1.93
Area, % change	...	-7.38	-6.15	-6.04	...	-1.94	-1.31	-1.38
Extractables, %	3.77	2.93	2.72	3.32	5.50	5.89	5.87	5.33
Volatiles, %	0.51	0.88	1.19	2.57	0.15	0.12	0.10	0.06
		Percent retention				Percent retention		
Tensile at fabric break	35.85 ppi	125[e]	135	139	...	...	...	...
Tensile at ultimate break	1728 psi	153[e]	151	161	2620 psi	99[e]	93	96
Elongation at break	243%	61[e]	52	52	665%	96[e]	96	95
S-100	938 psi	199[e]	232	248	932 psi	109[e]	107	119
S-200	1550 psi	...	...	...	1018 psi	109[e]	107	116
Tear strength (Die C)	...	...	...	...	8.56 lb	107[e]	109[e]	98
		Change in points				Change in points		
Hardness	77A	-0A	-1A	-3A	89A	+0A	-2A	+0A
	...	...	...	...	32D	...	...	+6D

Continued

APPENDIX K. (Continued)

Polymer	Neoprene				Neoprene			
Compound type^b	XL				XL			
Fabric, type	...				...			
Thread count, epi	...				...			
Nominal thickness, mils	34				60			
Liner number^d	43				82			
Exposure time, days	...	343	745	1231	...	343	745	1231
Weight, % change	...	-1.86	-2.96	-3.11	...	-0.53	-1.17	-1.31
Area, % change	...	-2.29	-1.96	-3.86	...	-1.32	-1.31	-2.32
Extractables, %	13.69	11.71	10.98	9.93	13.43	11.97	12.05	11.45
Volatiles, %	0.45	0.66	0.79	1.03	0.19	0.47	0.53	0.76
		Percent retention				Percent retention		
Tensile at fabric break	...	...	...	...	...	...	...	...
Tensile at ultimate break	1785 psi	94^e	88	88	1755 psi	95^e	93	92
Elongation at break	320%	76^e	70	63	400%	79^e	66	63
S-100	460 psi	146^e	170	184	383 psi	140^e	175	188
S-200	1038 psi	135^e	144	149	790 psi	134^e	159	168
Tear strength (Die C)	5.41 lb	80^e	86^e	86	11.13 lb	84^e	87^e	82
		Change in points				Change in points		
Hardness	57A	+10A	+11A	+14A	57A	+8A	+9A	+12A

Continued

APPENDIX K. (Continued)

Polymer	Polyester				Polyvinyl chloride				Polyvinyl chloride			
Compound type[b]	CX				TP				TP			
Fabric, type	...				...				...			
Thread count, epi	...				...				...			
Nominal thickness, mils	7				30				33			
Liner number[d]	75				11				59			
Exposure time, days	...	343	745	1231	...	343	745	1231	...	343	745	1231
Weight, % change	...	-2.50	-8.32	-6.23	...	-1.36	-8.40	-15.61	...	-1.45	-6.66	-10.31
Area, % change	...	-1.29	-1.00	-1.42	...	-2.90	-6.09	-10.28	...	-3.92	-7.39	-10.75
Extractables, %	2.74	[g]	3.42	3.92	33.90	33.86	30.33	26.27	35.86	34.94	30.31	27.78
Volatiles, %	0.26	0.35	0.12	0.13	0.15	0.18	0.45	0.42	0.31	0.17	0.23	0.13
		Percent retention				Percent retention				Percent retention		
Tensile at fabric break	...	...	...	...	...	...	...	...	...	...	...	...
Tensile at ultimate break	6768 psi	75[e]	66	70	2878 psi	96[e]	96	97	2558 psi	96[e]	107	111
Elongation at break	575%	89[e]	84	83	357%	100[e]	91	77	375%	96[e]	93	86
S-100	2585 psi	104[e]	107	110	1420 psi	107[e]	127	161	995 psi	123[e]	161	185
S-200	2733 psi	100[e]	103	107	2013 psi	100[e]	111	128	1580 psi	107[e]	132	145
Tear strength (Die C)	5.92 lb	89[e]	89[e]	103	11.20 lb	97[e]	123[e]	134	9.89 lb	102[e]	135[e]	144
		Change in points				Change in points				Change in points		
Hardness	93A 45D	-6A ...	-1A ...	-1A +7D	80A ...	+0A ...	+2A ...	+7A ...	73A 26D	+3A ...	+10A ...	+13A +13D

[a] Extractable content measured in accordance with ASTM D3421, modified so as to allow for suitable solvent of polymer type; volatile content is percent loss in weight after 2 hours at 105°C; tensile values measured in accordance with ASTM D412 and D638, using a small tabend dumbbell with a 0.25 in. neck and tested at 20 ipm. Tear strength measured in accordance with ASTM D624. Reported values for both tensile and tear are average of machine and transverse directions except where otherwise noted. Hardness measured in accordance with ASTM D2240.

[b] XL = crosslinked or vulcanized; TP = thermoplastic; CX = crystalline.

[c] Thread count in the machine and transverse directions, respectively.

[d] Matrecon liner serial number; R = fabric reinforced.

[e] Retention of machine direction only.

[f] Bulk of lining material's strength is in the nylon fabric. The butyl coating over the fabric tended not to fail catastrophically, and no useful value could be obtained for tensile at ultimate break.

[g] Extractable content not determined because of lack of material.

APPENDIX L

VOLATILES TEST OF UNEXPOSED POLYMERIC LINING MATERIALS
(Matrecon Test Method 1 - October 1982)

Scope

This test is to be performed on unexposed membrane liner materials.

Significance

This test can be used to determine the volatile content of an unexposed sheeting, including water, volatile oils, and solvents. Nonvolatile dissolved or absorbed components of a specimen will be determined by the extractables test which is run after the volatiles have been removed (see Matrecon Test Method 2). The volatile content should be determined as soon as possible after the liner has been received. By identifying the orientation of the disk with respect to the sheeting at the time it was died out, the grain of the sheeting can be established.

Definitions

Volatiles are the fraction of weight lost by a specimen during the specified heating process described below.

Apparatus

- Two-inch interior diameter circular die.
- Analytical balance.
- Air-oven.

Test Specimen

Two-inch diameter disks died out of the sheeting, as received.

Number of Test Specimens

All determinations should be run in duplicate.

Procedure

1. Draw a line on the sheeting to mark "grain" or machine direction. If the "grain" is unknown, draw a random straight line on the sheeting.

2. Die out a 2-in. diameter disk so that the lines fall approximately in the middle of the specimen.

3. Weigh specimen in tared, closed container to the nearest 0.0001 g. Record weight "as received weight."

4. Dry specimen out on Teflon screen for two hours at 105±2°C.

5. Cool in desiccator for 20 minutes.

6. Weigh on analytical balance to 0.0001 g; record as the "oven dry weight."

7. Measure diameters in machine and transverse directions. Record to 0.001 inches.

8. If machine direction is unknown, find and record largest and smallest diameters of disk. Mark small diameter as machine direction on disk as shown in Figure L-1. Use the dried disk to determine the orientation of the sheeting from which it was removed.

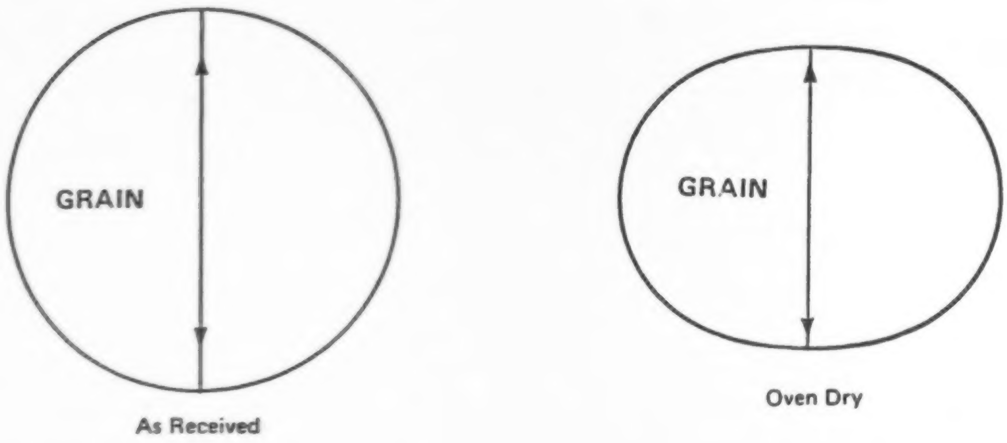

Figure L-1. Machine direction determination.

9. Retain specimens for additional testing, e.g. specific gravity, thermogravimetry, extractables, etc.

Calculations

Calculate the percent volatiles as follows:

$$\text{Volatiles, \%} = [(A-B)/A] \times 100$$

where:
A = grams of specimen, "as received weight"
B = grams of specimen, "oven dry weight"

Report

1. Identification of sheeting.
2. Result of above calculation of volatiles.

APPENDIX M

TEST FOR THE EXTRACTABLE CONTENT OF UNEXPOSED LINING MATERIALS
(Matrecon Test Method 2 - October 1982)

Scope

This procedure covers the extraction of plasticizers, oils, and other solvent-soluble constituents of polymeric lining materials with a solvent that neither decomposes nor dissolves the polymer.

References

This procedure generally follows ASTM D3421, "Extraction and Analysis of Plasticizers Mixtures from Vinyl Chloride Plastics". See also ASTM D297, "Rubber Products-Chemical Analysis," Paragraphs 16-18.

Significance

The extractable content of a polymeric lining material can consist of plasticizers, oils, or other solvent-soluble constituents that impart or help maintain specific properties such as flexibility and processability. During exposure to a waste, the extractables content may be extracted out by the waste resulting in a change in properties. Another possibility is that during exposure the material could absorb nonvolatilizable, in additon to volatile, constituents from a waste. Measuring the extractable content of unexposed lining materials is, therefore, useful for monitoring the effect of an exposure on a lining material. The extract and the extracted liner obtained by this procedure can be used for further analytical testing. e.g. gas chromatography, infrared, ash, thermogravimetry, etc. for fingerprinting the liner.

Apparatus

- Aluminum weighing dishes.
- Analytical balance.
- Air-oven.
- Soxhlet extractor (or rubber extraction apparatus).
- Extraction thimbles.

- 500 mL flat-bottomed flask (or 400 mL thin-walled Erlenmeyer flask if rubber extraction apparatus is used).

- Hot plate or steam plate.

- Boiling beads.

- Cotton wool.

- Aluminum foil.

Note: The rubber extraction apparatus may be substituted for the Soxhlet with all polymers except PVC and CPE. An appropriate reduction in sample size and solvent volume must be made. The metal condensers of the rubber extraction apparatus are corroded by HCl which is produced during extraction of the PVC and CPE.

Reagents

Table M-1 lists the recommended solvents for the extraction of membrane liners of each polymer type.

TABLE M-1. SOLVENTS FOR EXTRACTION OF POLYMERIC MEMBRANES

Polymer type	Extraction solvent
Butyl rubber (IIR)	Methyl ethyl ketone
Chlorinated polyethylene (CPE)	n-Heptane
Chlorosulfonated polyethylene (CSPE)	Acetone
Elasticized polyolefin	Methyl ethyl ketone
Epichlorhydrin rubber (CO and ECO)	Methyl ethyl ketone or acetone
Ethylene propylene rubber (EPDM)	Methyl ethyl ketone
Neoprene	Acetone
Nitrile rubber (vulcanized)	Acetone
Nitrile-modified polyvinyl chloride	2:1 blend of carbon tetrachloride and methyl alcohol
Polyester elastomer	Methyl ethyl ketone
High-density polyethylene (HDPE)	Methyl ethyl ketone
Polyvinyl chloride (PVC)	2:1 blend of carbon tetrachloride and methyl alcohol
Thermoplastic olefinic elastomer	Methyl ethyl ketone

Note: Because lining materials can be sheetings based on polymeric alloys which are marketed under a trade name or under the name of only one of polymers, this list can only be taken as a guideline for choosing a suitable solvent for determining the extractables. Once a suitable solvent has been found, it is important that the same solvent be used for determining the extractables across the range of exposure periods.

Sample size

If using the Soxhlet extractor, about five grams of devolatilized material are needed per extraction. If using the rubber extraction apparatus, about two grams are needed. All extractions should be run in duplicate.

Procedure

1. Cut the sample into cubes no larger than 0.25 in. on a side.

2. Weigh sample into an aluminum weighing dish and dry in moving air at room temperature for more than 16 hours.

3. Place in air-oven for two hours at 105±2°C. Weigh the sample.

4. Weigh the sample into a tared extraction thimble. Plug small thimbles with a piece of cotton wool to prevent the pieces from floating out of the thimble. (Large thimbles are tall enough to stay above the level of the liquid.)

5a. For PVC and CPE materials: Add 200 mL of extraction solvent to the 500 mL flat-bottom distillation flask. Add boiling beads to reduce bumping.

5b. For other materials: Dry and preweigh a thin-walled Erlenmeyer distillation flask. Add 200 mL of extraction solvent to the flask.

6. Place the thimble in the extractor barrel, put the condensor in place, and run the extraction a minimum of 22 hours. Aluminum foil can be wrapped around the extractor and flask to increase the distillation rate.

7a. For PVC and CPE materials: When the extraction is complete, rinse all the solvent from the extractor barrel into the distillation flask. Decant the solvent from the flask into a dried, tared 500 mL Erlenmeyer flask and then evaporate on a steam bath with filtered air. Place the flask in an oven at 70±2°C and dry two hours. Hold the extract for further testing e.g. gas chromatography and infrared.

7b. For other materials: When the extraction is complete, rinse all the solvent from the extractor barrel into the distillation flask. Evaporate the solvent from the flask on a steam bath with filtered air. Place the flask in an oven at 70±2°C and dry two hours. Hold the extract for further testing.

8. If the extract contains constituents which may volatilize during the evaporation procedure or is to be used for further analysis, heat the flask with extract in solution on a 70°C hot plate or a steam plate to near dryness. Complete evaporation of solvent in vacuum oven at 40°C.

9. Remove extracted liner from the thimble after excess solvent is removed and place in a tared aluminum weighing dish. Heat to constant weight at 105°C. Extracted PVC specimens cannot be dried to a constant weight at 105°C when they are extracted with a blend of CCl_4 and CH_3OH because, once the stabilizer additives have been removed by extraction, the polymer undergoes slow decomposition with HCl loss. It is recommended that the sample be dried 72 hours at 105°C. Hold the extracted liner for further testing.

   Note: In cases where the extracted specimen sticks to the extraction thimble, the extraction thimble should be dried to constant weight at 70°C before the extraction and the weight recorded as the true weight of the thimble. After the extraction, the extracted liner can be dried to a constant weight in the thimble.

Calculations

Calculate the percent volatiles as follows:

Volatiles, % = [(A-B)/A] x 100

where:

A = grams of specimen, as received
B = grams of specimen after 2 hours at 105°C

Calculate the percent extractables as follows:

Extractables, % = (B/A) x 100

where:

A = grams of specimen
B = grams of dried extract

Note: In cases where the extract may contain some constituents which volatilized while the extraction solvent was evaporated, the percent extractables should also be calculated as follows:

Extractables based on loss from specimen, % = [(A-B)/A]

where:

A = grams of specimen
B = grams of extracted liner

Report

1. Identification of sheeting.
2. Extraction solvent.
3. Volatiles.
4. Extractables.
5. Extractables based on loss from specimen, if calculated.

APPENDIX N

PROCEDURE FOR THE ANALYSIS OF UNEXPOSED POLYMERIC MEMBRANES BY PYROLYSIS

Apparatus

1. Dry and oxygen-free N_2 gas.
2. Air or oxygen.
3. Size 00 000 pyrolysis boat.
4. Quartz or Vycor tube.
5. Rotameter with a flow capacity of at least 500 mL/minute.
6. Thermocouple and read-out usable at 500 - 600°C.
7. Bunsen burner.

Procedure

1. Heat clean size 00 000 pyrolysis boats at 600°C for 10 minutes to drive off contamination.
2. After cooling, weigh the boats (about 0.4 g each) to the nearest 0.0001 g.
3. Place 0.02 to 0.04 g of sample cut with a razor blade into the boat. Keep the volume small to reduce boil out.
4. Flush a hooded Vycor tube with dry, oxygen-free nitrogen at about 500 mL/minute.
5. Set a medium hot Bunsen burner about 2-3 in. below the tube and adjust the heat until a thermocouple in the center of the tube registers 500 - 550°C.
6. Insert the boat in the tube end opposite the flow and push slowly to about 1-2 in. from the hot zone. Advance sample cautiously allowing boiling and gas evolution to begin slowly. Nearly all gas evolution should be complete before final insertion into the hot zone.
7. Heat at 500 - 550°C for at least 5 minutes after all visible changes are complete.

8. Push sample out of hot zone and allow to cool in the nitrogen stream.

9. Weigh and record on data sheet.

10. Flush the tube with O_2 at 500 mL/min and insert sample directly into the 500 - 550°C hot zone. The sample should remain until all visible black is gone and the sample is at least gray in color (it will often be white).

11. Cool the sample in the gas stream and reweigh.

12. Calculate the composition as indicated in the sample calculation.

13. Two samples should be run. If results are not within 2% of each other, run two more samples.

14. Boats which will not burn clean may be treated with warm, concentrated nitric acid, rinsed with water, and then brushed to remove residues. Boats should be heated to pyrolysis temperature before reweighing for use.

Sample Calculation

The data given are for a PVC compound. All values are the average of two determinations.

Data:
A = % loss on pyrolysis at 550°C under N_2 = 81.3
B = % further loss on pyrolysis at 550°C under O_2 = 11.2
C = % loss on solvent extraction = 33.9
D = % carbonaceous residue (from Table below) = 4.9

Calculations:
Organic polymer = (A+D-C) = 52.3%
Monomeric organic additives (extractables) = C = 33.9%
Carbon black = (B-D) = 6.3%
Inorganic filler (ash) = (100-A-B) = 7.5%
Total 100%

TABLE N-1. CARBONACEOUS RESIDUE
FOLLOWING PYROLYSIS UNDER N_2 AT 550°C

Polymer	Weight, %	
	Matrecon	Wake[a]
Neoprene	13.9%	13.9%
Polyvinyl chloride	4.9	5.9
Chlorosulfonated polyethylene	0.5	...
Butyl rubber	...	0.10

[a] Source: Wake, 1969, p. 141.